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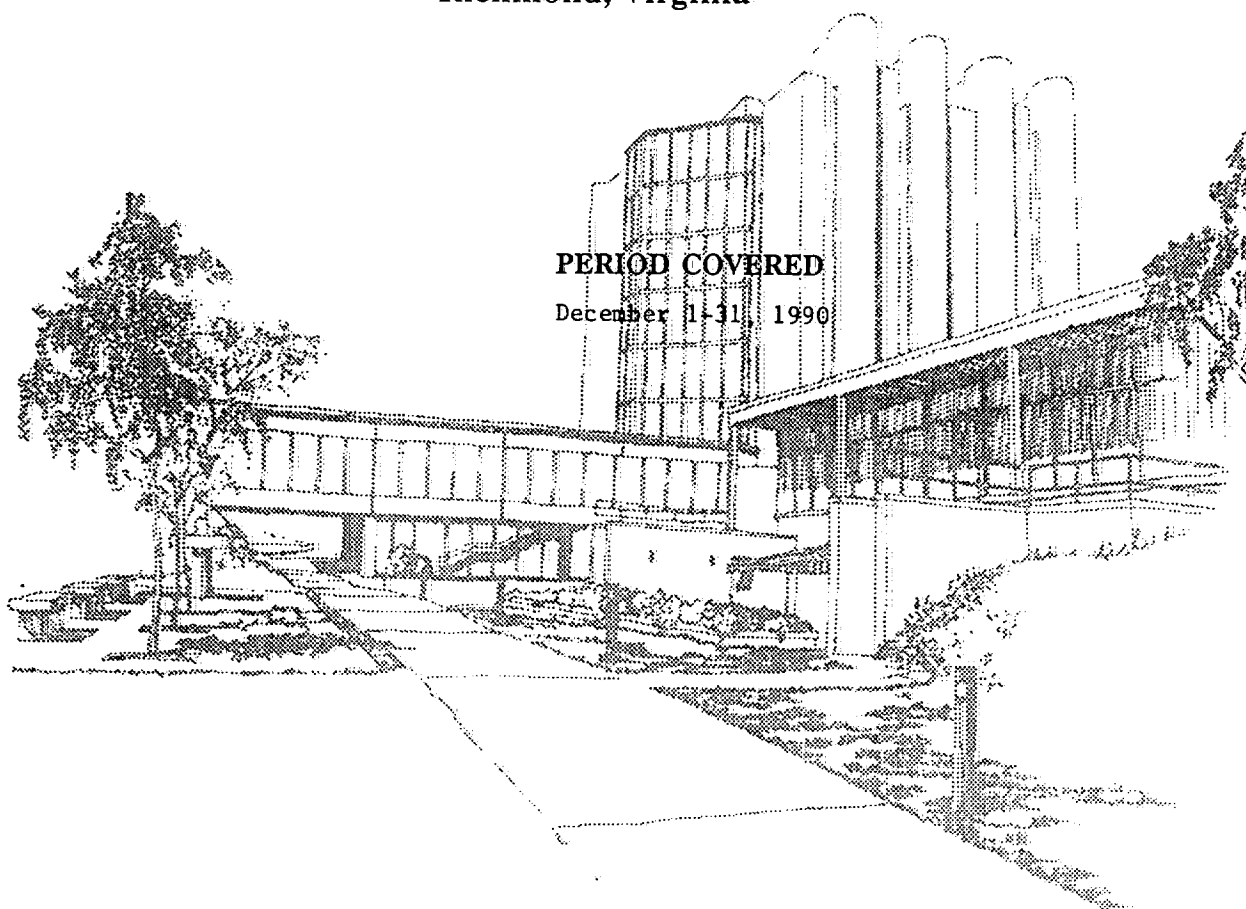
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**PHILIP MORRIS USA**  
Research Center  
Richmond, Virginia

**PERIOD COVERED**

December 1-31, 1990



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\* N/R = No Report

**PROJECT NUMBER :** 2108  
**PROJECT TITLE :** New Product and Filter Technology  
**PROJECT LEADER :** W. T. Callahan  
**PERIOD COVERED :** December, 1990

## **I. PM PAPER AND FILTER DEVELOPMENT**

- A. Objective:** To characterize web-type filter materials and develop subjectively acceptable products with them.
- B. Status:** Subjective results were received from the evaluation of Tela paper coated with 20% CA which indicated a positive response compared to uncoated Tela paper. James River Corporation was contacted and is investigating a technique for production of larger quantities of the CA coated material.

Sample cigarettes were hand-made with the latest James River produced PM papers. Models with Tela paper, James River 100% wood paper, 75% CA/25% softwood paper, and CA tow as mono filters were submitted for subjective evaluation. The 75/25 filter tested favorably to the CA tow control.

Sample hand-sheets with acetylated cellulose fiber (Batch 1,3,5 from Forest Products Lab) were received from Paper Technology personnel. The sheets were made into hand-made filters and cigarettes for subjective screening to determine the desired degree of acetylation.

Filtration efficiency data on the latest (November 8, 1990) James River web filters were received. This data showed similar efficiencies for like models produced on August 16, 1990 at James River. The efficiencies are comparable to paper per unit RTD. Neither Cellulon nor Buckeye cellulose yielded an increase in total efficiency per unit RTD versus TELA paper. Additional runs to produce larger quantities of PM web at James River are scheduled for January 3-4, 1991.

DAP and  $K_3PO_4$  solutions, CEL and Burley solubles were coated onto Tela paper hand sheets by Paper Technology personnel. Three levels of addition were obtained: approximately 3, 6, and 9%. The sheets have been made into filter rods via the bench top corrugator. Hand made cigarettes will be prepared for subjective evaluation to determine possible improvements to the taste of paper filters.

## **II. MACHINERY DEVELOPMENT**

- A. Objective:** Develop or modify manufacturing equipment to support Product Development efforts.
- B. Status:** The utilities and guarding for the Strip Application Unit (SAU) have been installed.

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The Strip Application Unit (SAU) has been used to produce bobbins of paper with standard cigarette base paper and bands of either capacitor paper (all cellulose - 35 g/m<sup>2</sup>) or Trim outer wrap (47 g/m<sup>2</sup>). Further evaluations of adhesives, adhesive patterns, and paper combinations are planned.

Tests have begun to use the bench top plug maker to produce 6mm diameter filter plugs. The intent is to develop the capability to replace the inner core of concentric filters produced at American Filtrona with various filtration materials of interest. Further modification of the bench top unit is necessary and parts are being redesigned.

### III. NOVEL FILTERS AND FILTER MATERIALS

- A. **Objective:** To investigate novel filters or filter materials for potential applications where increased efficiency, taste response or a novelty is needed.
- B. **Status:** Sample filters were produced for evaluation in the BOLD 100's core. PM paper (75% CA/25% wood fiber) and Celanese CA web were evaluated and PM web will be further pursued as a core material.

Base concentric filter rods for the BOLD 100's combiner at Anderson Machine were produced at American Filtrona and utilized for machine set up.

Component rods for a BOLD KS Paper Core Concentric (PCC) recessed filter model were produced at American Filtrona. The filters will be combined at Stockton Street, and cigarettes will be produced.

### IV. ALTERNATIVE PLASTICIZERS

- A. **Objective:** Qualify alternative plasticizers which offer a product advantage.
- B. **Status:** Flavor Development subjectively compared four triethyl citrate (TEC) plasticized Marlboro KS cigarette models against a triacetin (TA) plasticized control. The four TEC models consisted of two models plasticized with untreated TEC and two models plasticized with TEC treated with 10% PG with similar tar and nicotine deliveries. Flavor Development found the untreated TEC model most similar to the control. The Test and Control models were submitted to PED for more detailed subjective evaluations by an in-house walk-in panel.

### V. CARBON STUDIES

- A. **Objective:** Conduct fundamental studies of carbons and carbon filtered cigarettes to better understand their performance.
- B. **Status:** An investigation is under way to determine the effect of tobacco volatiles upon carbon activity and subjective response. Lark Super Lights and PM Lights cigarette models with and without carbon were made in Semiworks and submitted to

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Flavor Development for analyses of the tobacco head space. The head space analyses will be done after 2 weeks, 1, 2, 3, 4, 5, and 6 months.

## VI. MENTHOL TECHNOLOGY

- A. **Objective:** To develop alternative mentholation techniques which offer a product advantage.
- B. **Status:** After five weeks of desert room aging, mentholated dual CA/paper filters continue to show an improvement in stability over mentholated mono-CA filters. Additional models are planned which will have equivalent initial menthol-in-smoke values for subjective testing.

## VII. PRODUCT DEVELOPMENT SUPPORT

- A. **Objective:** To provide cigarette/filter design, modelling and testing support for major new product development programs.
- B. **Status:** A meeting was held between R&D and Manufacturing Services personnel to discuss the status and plans for the continued development of the porous combining wrap for Lark. The most promising porous combining wrap to date is an Ecusta material with a modified hot melt adhesive (EVA coating on the felt side). Combining and tipping trials are in progress. Several Kimberly-Clark models will be evaluated in early January.

A meeting was held with Manufacturing Services and M/C Quality Audit personnel to enlist QA support for the evaluation of tipping and filter defects on Lark cigarettes produced with test combining wraps. Test cigarettes currently on-hand were submitted for evaluation. Data from the evaluations will be communicated to the material suppliers.

Preliminary experimentation has begun to identify CA/water emulsion systems that could be employed for application to paper web to improve its subjective quality. Dispersions of CA flake in water appear to be capable of attaining an increase of about 10% maximum uptake onto TELA web. Previous testing has shown that around 25% application rate is necessary to achieve the desired subjective response. Multi-component systems will be investigated to improve the amount of CA applied.

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**PROJECT NUMBER :** 2305  
**PROJECT TITLE :** Applied Flavor Investigation  
**PROJECT LEADER :** J. W. Swain  
**PERIOD COVERED :** January, 1991

## **I. PROCESS SUPPORT**

- A. Objective:** To develop and evaluate process modifications to maintain subjective and physical quality.
- B. Status:** POL 3643 testing RLTC from the evaporator upgrade at Park 500 (Run 2) in Marlboro is in the field. Other tests in progress include reduced humectant levels in RCB and RL's in addition to stem level tests in RL's.

Large-scale Semiworks trials of the RL's with increased solubles levels compensating for reduced humectants have been requested. Physical testing through the primary and make/pack areas is planned in addition to subjective evaluations.

**C. Plans:**

|   |               |
|---|---------------|
| Initiate Large-Scale Tests of<br>Reduced Humectants | January, 1991 |
|---|---------------|

|   |               |
|---|---------------|
| Complete Tests of Stem Level<br>in RL's | January, 1991 |
|---|---------------|

## **II. OPERATIONS SUPPORT**

### **PROJECT GRAIN**

- A. Objective:** To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.
- B. Status:** Reductions of 20% alcohol level have shown promise in aftercuts without sonolation or adjustment in volume. Further testing and equipment evaluations are planned to optimize higher reductions in aftercuts. While current tests of the Burley Top Casing with reductions of 50% alcohol have shown no differences in Marlboro, further internal tests of 75% and 100% reductions are planned prior to POL tests.

The trip to the sonolator vendor (Sonic) will be rescheduled during January when the Process Engineer can attend. A quote has been received for a laboratory model sonolator from Sonic Corporation.



**C. Plans:**

|   |               |
|---|---------------|
| Initiate Large-Scale Tests<br>of 75% and 100% Reductions<br>of Alcohol in BTC | January, 1991 |
|---|---------------|

**III. DRY FLAVOR REPLACEMENT**

A. **Objective:** To develop, evaluate and establish specifications for dry flavor replacements.

B. **Status:** Implementation of the flavor substitutions in RLB is planned for January, 1991. Flavor transmittals have been made detailing preparation, analytical procedures and specifications. Flavor sufficient for the first month of production has been made and is under evaluation prior to preparation of the solid extract at the Flavor Center.

**C. Plans:**

|   |                        |
|---|------------------------|
| Flavor Replacement<br>Implementation at<br>Park 500 | January, 1991          |
| Analytical and Subjective<br>Flavor Batches         | January, 1991 Tests of |

**IV. CAST LEAF PROGRAM**

A. **Objective:** To develop and evaluate flavor systems for cast sheet utilization.

B. **Status:** Spanish ASTA trials were completed for qualification tests in Spanish Marlboro. Large-scale Semiworks trials have been planned to evaluate ASTA substituted for partial and total blend levels of RCB. Flavor systems including increased levels of DAP continue to be evaluated for SIVA to replace RL's in Spain. Modification of the original flavor system showed slight subjective differences. Further tests are in progress.

**C. Plans:**

|                                      |                |
|--------------------------------------|----------------|
| Complete ASTA Qualification<br>Tests | February, 1991 |
| Recommend Flavors for SIVA<br>Tests  | January, 1991  |

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**PROJECT NUMBER :** 2306  
**PROJECT TITLE :** Marlboro Standardization/International Support  
**PROJECT LEADER :** W. R. Bell  
**PERIOD COVERED :** January, 1991

## **I. MARLBORO STANDARDIZATION**

- A. **Objective:** Analytical and subjective evaluations of production Marlboro KS/LS.
- B. **Status:** Data from POL tests, Marlboro Standardization Smoking Panel and Analytical is being reviewed and will be compiled and reported to the Marlboro Standardization Group.
- C. **Plans:** Compile analytical and subjective data from Standard VIII for the Standardization Group.

## **II. MARLBORO EXTRA LIGHTS, MARLBORO MEDIUM/MILDS**

- A. **Objective:** To develop a Marlboro line extension in 8-9 mg (Extra Lights) and/or 12-13 mg (Medium/Milds) using cork tipping.
- B. **Status:** Ad Pack tests were shipped to New York in November for both Medium/Milds and Extra Lights. POL 0361 cigarettes using Blend 259 and Marlboro Ultra Lights flavor system have been produced in Semiworks. This product is targeted to deliver 9 mg tar (FTC) and will be subjectively analyzed pending C.I. results. All casings and aftercut were analyzed by Basic Flavor Investigation.

Six models using Blend 259 and Merit Blend with Marlboro Ultra Lights aftercut, modified Marlboro aftercut and a new Marlboro Extra aftercut were produced in the Semiworks. These models are for internal evaluation of blends and flavors.

- C. **Plans:** Conduct subjective evaluations on new models while awaiting POL results.

## **III. DOMESTIC PANEL**

- A. **Objective:** Provide subjective evaluations (rod aroma and smoking characteristics) for domestic markets.
- B. **Status:** Fifteen (15) panels were conducted during this reporting period.

## **IV. INTERNATIONAL SUPPORT**

- A. **Objective:** Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.

- B. **Status:** Seventeen (17) panels were completed during this reporting period.

V. **MARLBORO (KOREA)**

- A. **Objective:** Develop a reduced tar Marlboro cigarette to better compete in the Korean market.
- B. **Status:** The five models with different casings and aftercuts on GCC blends have been subjectively screened against the control. Three models have been selected as having a "softer" subjective profile as compared to the control. Two models will be selected as "best" candidates for a future Seoul Consumer Panel.

VI. **PROJECT VICEROY (PANAMA)**

- A. **Objective:** Develop a prototype to compete with Viceroy (BAT) in Panama.
- B. **Status:** Two experimental aftercuts have been developed to compete with Viceroy in Panama. Preblends for the aftercuts have been compounded and analyzed by the Flavor Center and Flavor Development. These have been shipped to Panama for evaluation.

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**PROJECT NUMBER :** 2307  
**PROJECT TITLE :** Basic Flavor Investigation  
**PROJECT LEADER :** R. W. Hale  
**PERIOD COVERED :** January, 1991

**I. ANALYTICAL SUPPORT**

**A. Objective:** To provide analytical support for activities related to development and application of flavoring materials.

**B. Results:**

1. Seven aftercut flavors were analyzed for anethole, vanillin, propylene glycol, glycerin and ethanol.
2. Three licorice samples were analyzed for propylene glycol, glycerin, sucrose, fructose, glucose, maltose, water and glycyrrhizic acid.
3. Four Brazilian menthol samples were analyzed by GC and GC/MS to obtain baseline data for future reference of this product.
4. Three St. Johns Bread samples for "Dry Flavor Replacement" were analyzed for sucrose, glucose, fructose and propylene glycol.
5. GC profiles were obtained for three SFC-BT casings and also analyzed for propylene glycol and ethanol.
6. Three ES casings were analyzed for sucrose, glucose, fructose, glycerin and APA.
7. Six samples of Burley Spray, Burley Top and Bright casings (2 each) were analyzed using the standard procedures for these samples.
8. Twenty-two samples of filler were analyzed for sucrose, glucose and fructose.
9. Twenty cigarette samples were analyzed using the headspace technique, vanillin and menthol were also determined for some of these cigarettes.
10. Two samples of 04-358 were analyzed for ethanol, water and propylene glycol.
11. International Support:

Headspace and analytical profiles were completed on 6 cigarettes, 88 Lts, 88 Gold, Pine Tree Golden Lts, Mt. Holla, Ballroom Flavor, Mild Seven Lts.

Analyzed two Burley Top samples (Amethyst Project) for water, propylene glycol and propylidene phthalide.

## II. FLAVOR INVESTIGATION

A. Objective: To develop new basic and applied flavor technology in support of new product development objectives.

B. Results:

1. The identification of the components responsible for the off odor in triacetin and glycerin is ongoing.
2. Evaluation of water soluble flavor/cyclodextrins complexes is in progress.
3. Evaluation of monoammonium glycyrrhizinate as an emulsifier for PM aftercut flavor system is in progress.

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**PROJECT NUMBER :** 4016  
**PROJECT TITLE :** Paper Technology  
**PROJECT LEADER :** S. D. Baldwin  
**PERIOD COVERED :** December, 1990

## **PAPERMAKING PROCESS DEVELOPMENT (B. Rogers)**

### **I. EXPERIMENTAL PAPERS (B. Rogers, N. Gautam)**

**A. Objective:** Develop proprietary cigarette papers for low sidestream smoke or product improvement.

**B. Results:** Due to the current backlog of handsheets for the Low Sidestream project, a number of requests were transferred to The University of Maine. Handsheet procedures were developed at Maine that duplicate the methods used at Philip Morris. A total of six handsheet requests were completed during the first week of production. All sheets will be coated at Philip Morris.

Handsheet facilities are scheduled to be moved during the first week of January to Semiworks. In addition, a second 12" square handsheet mold has been ordered to facilitate handsheet production. The new lab should be fully operational by the second week of January.

Three areas of process development are being pursued at the University of Maine to improve both sheet appearance and strength. The object is to develop the capability to further reduce the fiber length of flax at equal freeness. Two new types of flax have been obtained (Kimberly-Clark and Type B flax from Danforth) for evaluation early next year. The condition of the beater at Maine will also be evaluated in January to determine if this unit's refining capabilities have deteriorated. New disc refining plates have been ordered.

**C. Plans:** Evaluate process changes at University of Maine. Install second handsheet mold in new laboratory facilities.

### **II. BANDED PAPERS (B. Rogers, N. Gautam, J. Ryder, S. Baldwin)**

**A. Objective:** Incorporate cross directional bands of fiber and/or filler in cigarette papers in order to vary cigarette burn rate.

**B. Results:** Papers were produced with 5 g/m<sup>2</sup> Cellulon applied in the banded regions. Partial bands, bands composed of spots, and spotted papers were produced, all with an inherent porosity <1 Coresta in the banded region. Bands composed of <5 g/m<sup>2</sup> Buckeye Cellulose expanded fibers were ineffective in modifying burn rate since the banded section's porosity was 5 Coresta.

Quantities of Cellulon were cleaned by an experimental process in an effort to make this material more suitable for use. Samples have been submitted for nitrogen and

NMR analyses and to make banded handheets. The color of the cleaned material is much whiter, and the material has no malodor, as the raw Cellulon has.

Extrusion technology was discussed with Nordson as a possible method of applying cross directional bands to either wet (wet end of a paper machine) or dry paper. The technology is currently available to apply cross direction bands on dry paper up to 16" wide at about 100 fpm (single nozzle). A short nozzle section was ordered and will be evaluated on paper (both wet and dry) with cellulosic containing slurries (Cellulon and Buckeye's expanded fiber).

One of the banded drums at Maine will be modified for use as a "rotogravure" type cylinder to evaluate this concept. The inside of the drum will be blanked off and a portion of the slots will be filled to reduce the volume. Maine will construct a slurry box and doctor blade that will be mounted on the outside of the drum. This apparatus should allow the application of bands with  $\sim 1 \text{ g/m}^2$  add-on.

Beloit Corporation's paper machine research facility was toured to evaluate their ability to scale up the Daubing Dandy concept. They appear to be a viable alternative to developing and testing the concept at either Philip Morris or another contractual research facility. Several possible design options were discussed. Beloit will prepare a list with cost and completion dates for each option by the middle of January.

The sheet properties resulting from use of a top wire drainage device for banded papers was modeled in the handsheet lab by constructing bilayer handsheets. The moisture level of the banded region was varied (6 to 48% solids) while maintaining the base sheet at a constant solids level (flax/carbonate sheet at 12.8% solids). Sheet bonding decreased and both porosity and caliper increased as the solids level of the banded region was increased. Apparently formation and drainage of the banded material on the handsheet mold increased this fraction's porosity. The increase in caliper and decrease in tensile is an indication that the banded region is not being incorporated into the base sheet.

### III. FILTER DEVELOPMENT SUPPORT (B. Rogers, N. Gautam)

- A. **Objective:** Support the development of a proprietary filter material in web form for utilization by Filter Technology.
- B. **Results:** The success of the CA/softwood and 100% softwood models is attributed to the long cellulosic fiber content a portion of which tends to remain intact during the filter making's corrugation process. All the sheets lint excessively which is attributed to the use of CMC on the Yankee (to adhere the sheet for creping). Future runs, scheduled for early January, will be directed at improving the strength of the CA/softwood model and reducing the degree of lint.

Additional filter webs were analyzed for surface area. Combinations of partially acetylated cellulose and Cellulon give the highest surface area ( $\sim 2.2 \text{ m}^2/\text{g}$ , above CA web at  $\sim 2.0 \text{ m}^2/\text{g}$ , standard deviation =  $0.01 \text{ m}^2/\text{g}$ ). Handsheets were also prepared

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with partially acetylated Cellulon, partially acetylated softwood and Cellulon for surface area analysis.

- C. **Plans:** Complete scheduled web runs at James River.

#### PAPER PRODUCT DEVELOPMENT (B. Goodman)

##### I. REDUCED SIDESTREAM CIGARETTES (B. Goodman, B. Floyd, S. Baldwin)

- A. **Objective:** Develop subjectively acceptable cigarettes with reduced sidestream.

B. **Results:**

**Trim:** Kimberly-Clark made a millrun of base papers at three different porosities, each with ~10.5% monopotassium phosphate added at their Ancram mill. Porosities were acceptable and phosphate levels were very close to target (~10% by our colorimetric analysis). Cigarettes were made in Louisville with papers at 11, 13, and 14.5 Coresta, measured with the 20 cm<sup>2</sup> clamp. The ventilation target of 66% was hit with the 0.25 cm pressure drop tipping paper suggested by Cigarette Development. Because of this, tar deliveries were lowered compared to the November run. Sidestream reductions ranged from 69 to 74%, and the highest tar delivery was 6.6 mg. Cigarettes from the December 11 Louisville run were screened for weight, RTD, and dilution and given to Cigarette and Flavor Development for subjective smoking. Test 2, the 12 - 13 Coresta paper with 10.4% phosphate was chosen as the preferred model. Test 2, the preferred model, gave 71% reduction. From these limited data, we are achieving a 70% visibility reduction at a 95% confidence level. There were no observed differences in the number of cigarettes going out (22%), and there was no staining behind the charline.

The 11 experimental Superslims models from the November run in Louisville were used to determine the effect of porosity, calcium carbonate level and phosphate level on visibility, tar and puff count. Good fits were obtained in each case. The most striking finding was that tar is directly related to calcium carbonate level.

- C. **Plans:** Return to Louisville to repeat the experiment using the additional bobbins of paper still on hand. That study will be designed in collaboration with John Tindall.

#### PAPER AND CELLULOSE DEVELOPMENTAL STUDIES (G. Bokelman, S. Tafur, S. Baldwin)

##### I. MAGNESIUM CARBONATE PAPERS (G. Bokelman, S. Tafur)

- A. **Objective:** To produce commercial quantities of a magnesium carbonate paper.

- B. **Results:** The first step of a two-step process developed for the grinding of Baymag magnesite was executed. The first step, involving impaction crushing from rocks to a coarse sand, was completed on 45 tons of magnesite at Industrial Crushing (Cambridge, Ohio). Prior to initiation of this work the plant lay out and the material

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composition of all relevant equipment were evaluated to help assure that there would be no contamination problems. In addition, an analyzed, compatible mineral was processed through this equipment just prior to the crushing of the magnesite to evaluate any potential for contamination. Nine tons of the crushed Baymag magnesite, representing two different particle size ranges, have been shipped to Midwest Custom Services, Inc. (Fort Wayne, Indiana) for the second step in the grinding process. The magnesite crushed at Industrial crushing in mid December was processed on both the pin mill (with tungsten-carbide liner) and in the jet mill (with composite tungsten-carbide liner). These magnesite samples were processed on the commercial scale equipment at no cost to Philip Morris, and will be analyzed for trace metal content prior to processing larger quantities of the material to be used for a mill run at Ecusta.

Analyses were obtained for K, Mg and Ca levels of the Ecusta mixed filler handsheets sized with  $K_3$  citrate/citric acid. Levels of solublized Mg and Ca were found in a water wash of the paper to suggest the citric acid readily reacted with the  $Mg(OH)_2$  and, to a lesser extent, the  $CaCO_3$  filler in the paper to form soluble citrates.

- C. **Plans:** Pending the results of the trace metal analyses, preparation of larger quantities of the ground magnesite is scheduled for the week of January 22.

## II. MECHANISTIC STUDIES (S. Tafur, G. Bokelman, S. Baldwin)

- A. **Objective:** To conduct research to elucidate mechanisms by which low sidestream papers act and to develop support for patent claims.
- B. **Results:** Samples of magnesite/ $CaCO_3$ / $Mg(OH)_2$  handsheets from Ecusta sized with  $K_3$  citrate/citric acid have been submitted for analyses to determine if soluble and insoluble forms of magnesium can be differentiated. Probable reaction of the citric acid with the  $Mg(OH)_2$  filler to form magnesium citrate may contribute to the improved subjectives.

The results of a Box-Behnken study conducted with the Solar Energy Research Institute (SERI) quantified the effect of potassium ion concentration, pH, and pyrolysis temperature on the cellulose and paper pyrolysis product slate. Differences in the product mix resulting from different additives to papers are believed to affect subjectives. The work was done using Molecular Beam Mass Spectrometry (MBMS) to evaluate the pyrolysis and combustion product mix for flax samples and high basis weight paper samples to which three levels of  $KH_2PO_4$  were added at three pH levels (3, 4.4, and 9). The pH 9 represents  $K_2HPO_4$ . Results showed that three main product slates were a direct function of pH, potassium ion concentration, and temperature.<sup>1</sup> Those three principal classes of compounds were anhydrosugars, aldehyde and carbonyl compounds, and furan-type compounds. The data generated to date by this work is considered by patent counsel to be sufficient to support our patent claims for PM 1393. This patent is for the high paper which will be used for the single wrap Virginia Slims Superslims. Additional data also indicates that there

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is an interaction between the phosphate and the calcium carbonate which affects the products formed when the paper is burned or pyrolyzed. The effect depends on the amount of oxygen present.

Other parallel experiments on a bacterial cellulose grown on  $^{13}\text{C}$ -enriched media have now shown definitive results. *Acetobacter xylinum* was grown on media that included glucose that was  $^{13}\text{C}$  labelled at the C-1 position, resulting in  $^{13}\text{C}$  levels of approximately 14%. This cellulose was pyrolyzed under both slow- and fast-heating conditions and the mass spectral results compared to unlabeled cellulose that was prepared in the same fashion. In the labelled sample, a higher peak was observed at  $m/z$  145, which indicates inclusion of the C-1 carbon into levoglucosan. No enhancement was observed for  $m/z$  61, which indicates that the glycolaldehyde does not come from the C-1 carbon in the anhydroglucose ring. Addition of 0.1% potassium as KOH did not yield any enhancement of  $m/z$  61. Again this indicates that the mechanism of formation of glycolaldehyde does not involve incorporation of the C-1 carbon. This is important mechanistic information concerning the mechanism of action of alkali metals on cellulose and paper pyrolysis and combustion<sup>1,2</sup>.

C. Plans: Evaluate the effect of  $\text{KH}_2\text{PO}_4$  on cellulose pyrolysis using the labelled material. Evaluate the effect of oxygen content on products of paper and cellulose pyrolysis and combustion.

D. References:

1. R. Evans, F. Abglevor, H. Chum, D. Chadick, J. Wooten, S. Baldwin, "On the Mechanism of Pyrolysis of Cellulose to Low Molecular Weight Compounds", paper presented at ACS National Meeting, Washington, D.C., August 29, 1990.
2. R. Evans, F. Abglevor, H. Chum, D. Chadick, J. Wooten, S. Baldwin, "New Approaches to the Study of Cellulose Pyrolysis", paper to be presented at ACS National Meeting, April, 1991.

2022201493

**PROJECT NUMBER :** 4030  
**PROJECT TITLE :** Export Product Development  
**SECTION LEADER :** J. N. Smith  
**PERIOD COVERED :** December, 1990

**I. Parliament (Japan)**

- A. Objective:** To evaluate the Parliament flavor system with and without citric acid.
- B. Status:** Parliament 100mm cigarettes were produced at Stockton Street on 12/5/90 for Danchi evaluation (E-N770). This test will evaluate the Parliament flavor system with and without citric acid at both 9mg and 12mg TIOJ tar. This test is scheduled to be shipped on 1/7/91 with results expected 2/14/91.
- C. Plans:** Similar Danchi testing of Parliament KS (E-N852) is planned for February.

**II. LTN PRODUCT (Korea)**

- A. Objective:** To develop a free-standing low tar and nicotine product to compete against 88 Lights in the Korean market.
- B. Status:** Results were received from the Ring SCP test (E-K0640) of two variations of the Ring flavor system with P-S-P filters at 8mg tar, PMSL with P-S-P filter, and 88 Lights. Among the overall panel and among 88 Lights smokers, the Ring A+ and Ring B models received significantly higher liking ratings than 88 Lights.
- C. Plans:** Further testing is planned with the Ring A+ flavor system and a series of filter designs including CA and CA/carbon concentric systems.

PROJECT NUMBER : 2304/4031  
PROJECT TITLE : U.S.A. Product Development  
SECTION LEADER : C. B. Altizer  
WRITTEN BY : G. N. Yatrakis and J. L. Spruill  
PERIOD COVERED : December, 1990

## **I. LOW TAR/HIGH FLAVOR**

### **PROJECT BOLD**

- A. **Objective:** Development of a 1mg 85mm and 2mg 100mm regular and menthol product competitive with Now and Carlton.
- B. **Status:** Samples were completed and submitted to C.I. and ARD using cigarette paper from four different lots of 10-028-A supplied from the Direct Materials Warehouse. Papers from Kimberly-Clark mill run using Albacar and Multifex chalk were received. The 85mm and 100mm reduced weight models using Blend 244 were completed. Samples were completed with Blend 253 (30/50 cpi) using burley spray and T.B.C. as the casing systems. The 30 vs. 50 cpi models and the "salt" casing models showed no improvement in puff count. Laboratory work is continuing to develop a new flavor system which reduces irritation and increases body and smoothness.

Models were supplied to P.E.D. for Internal Testing/Pack Handout Study. Samples included an improved 100mm model, Blend 244/-67 A/C and models using cased burley. Two menthol POLs (85/100mm) and two improved non-menthol models (85/100mm) close-out the end of the month.

- C. **Plans:** Develop new models for monadic POL testing.

## **II. PROJECT AMBROSIA**

### **AMBROSIA I**

- A. **Objective:** Develop a 23.0 circumference aromatic sidestream product as well as a 24.8 circumference 85mm and a 24.0 circumference 100mm and apply this technology to other products.
- B. **Status:** Analysis for A150 on the 24.8 prototypes has been completed. Results of the quantity of A150 was .15 mg/cigt., .26 mg/cigt. and .44 mg/cigt. These samples will be subjectively evaluated to determine the preferred target application rate for this model. C.I. results have not yet been completed.

Lab samples were made at National with two different batches of A150 on December 4 and 5. Adhesives samples were made with 5%, 7.5% and 10% A150. Samples were diluted to 700 cps and 500 cps, and will be monitored weekly for viscosity stability.

- C. **Plans:** Produce product with new production batch and finalize specifications.

### **AMBROSIA II**

- A. **Objective:** Develop K.S. and 100mm low smoke, low odor and low smoke/low odor prototypes.
- B. **Status:** A request has been submitted to make combination models using LSS paper with and without A-245 and A-150. Samples will also be made using Virginia Slims Superslims filler for subjective evaluation.
- C. **Plans:** Continue model making for improvements.

### **III. PROJECT MARLBORO**

- A. **Objective:** Design and develop Marlboro 83mm FTB products as possible line extensions.
- B. **Status:** Marlboro Red, Marlboro Lights, Marlboro 12-13mg and Marlboro 8-9mg have been made in 83mm Box/84mm S.P. and released for POL testing with close-out dates in early January. Marlboro Milds, Marlboro Mediums, Marlboro Extra Lights and Marlboro Ultra Lights were produced and released for Ad Pack, HTI and Market Research testing. Additional POL testing is scheduled for Marlboro Extra Lights with revised specifications to allow for a lower puff count.
- C. **Plans:** Evaluation of POL and Ad Pack test results.

### **IV. BRAND EXTENSIONS & MODIFICATIONS**

- A. **Objective:** Design and develop extensions and modifications to existing brand families.
- B. **Status:** New blends were received for Lark, Cambridge and Bristol with prototypes being requested in Semiworks. Development work continues on B&H King Size (full flavor/lights), Virginia Slims King Size (regular/menthol) and Parliament Lights 85/100's (regular/menthol).

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- C. Plans: Analytical and subjective evaluations of prototypes and monitoring of new brand start-ups.

2022201503

**PROJECT NUMBER :** 5001  
**PROJECT TITLE :** Packaging Studies  
**PROJECT LEADER :** B. S. Mait  
**PERIOD COVERED :** December, 1990

### **PACKAGING STUDIES**

A. **Objective:** Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. **Results:**

**Residual Printing Solvents in Packaging Materials:** Coordinated evaluation of the following items:

- Solvent Lacquer #RJC-7192
- Reynolds #19 foil with James River Dunn Tissue Surface Treatment

**Westvaco (Project Hairy):** A test plan outlining how lower fiber content would effect the integrity of flip top box material was established. This plan involves two test samples and one control.

**Penta-Pack:** The Lexan material was approved subjectively for the Penta-Pack display. The PET-G samples were rejected subjectively.

**Project Gold:** Two Findley pre-applied adhesives have been subjectively approved for use (#4408 and #4251/4408).

2022201504

PROJECT NUMBER : 1309  
PROJECT TITLE : Cast Leaf Development  
PROJECT LEADER : G. Gellatly  
PERIOD COVERED : December, 1990

#### CAST LEAF DEVELOPMENT

- A. **Objective:** Develop subjectively and physically acceptable reconstituted tobacco sheets for domestic and international application using cast leaf technology and proprietary binder systems.
- B. **Results:** **Cast Leaf Pilot Plant** - The dryer bids were issued and Somerset Technologies was selected on the basis of their expertise, low price, and satisfactory delivery time. This company can also supply the reverse roll coater and lecithin applicator. P&ID's and G.A. drawings have been finalized by Sirrine for approval by PM.

**Cast Leaf Development** - The heater below the Glenro heater belt was installed and is being evaluated. A significantly improved guar gum product has been produced in the Cast Leaf Laboratory using below belt heating only. Acceptable sheet using guar binder has been produced using two feedstocks (RL/BL blend and TSA blend using US components).

**Cadiz** - ASTA qualification trials were run in Cadiz. The plant operation was very steady and good quality sheet was produced. Three boxes of product were selected during each day of the five day trial (15 x 330 lbs = 4950 lbs) for shipment to USA for evaluation for subjective character and physical survivability. Cigarettes containing this product at 4 percent and 7 percent substitution of RCB in a Spanish MF blend will be made in the Semiworks in mid-January after the product has aged for one month. The ASTA product was doctored from the casting belt at 23 percent OV and dried to 13 percent OV in the drying cylinder. This post-drying crinkled the sheet to look like natural strip which prevented the settling in the packing case previously seen with the flat paper-like product.

The product was cooled after drying with refrigerated air to 72°F before packing. Four boxes were selected to follow the temperature changes and to study product darkening within the boxes with time. Four thermocouples were inserted in each box. The packout temperatures were much lower than experienced in the July and no significant change in case temperature occurred. Samples from the center and sides of the box were taken four days after packing, frozen, and hand carried to USA for examination for chemical change.

C. **Plans:**

1. Determine the conditions of OV, packout temperature, and packing density necessary to induce ASTA product darkening in the laboratory.
2. Rerun temperature and chemical profiles in packing cases in Cadiz during summer conditions.



1309-2

3. Continue binder and flavor development work to optimize the cast sheet formulation for USA and TSA.
4. Develop a trial schedule for SIVA trials in Cadiz 1Q/91. These trials will also evaluate the effect of the gum eductor and air removal from slurry on sheet quality.
5. Continue work with PM Engineering on design and installation of the Cast Leaf Pilot Plant.

**PROJECT NUMBER :** 1503  
**PROJECT TITLE :** Modified Smoking Materials  
**PROJECT LEADER :** W. A. Nichols  
**PERIOD COVERED :** December, 1990

**SIDSEAM ADHESIVE METERING SYSTEM**

- A. **Objective:** Evaluate a sideseam adhesive metering system for use in the Semiworks and as a prototype for Production.
- B. **Results:** A new controller was evaluated with the Intek low pressure drop flowmeter. An algorithm in the controller was designed to compensate for the lag in response time of the flowmeter transducer. Testing indicated some improvement at flowrates above 9cc/min. Based on these results a monitoring system rather than a closed loop control system is preferred for flowrates below 9cc/min. Redesign of the Kaymich glue gun making it a control element would be necessary for automatic control.
- C. **Plans:** The sideseam adhesive system will be installed in Semiworks and evaluated.

**PROJECT NUMBER :** 1806  
**PROJECT TITLE :** New Tobacco Processes  
**PROJECT LEADER :** T. C. Holland  
**PERIOD COVERED :** December, 1990

## **I. PRIMARY IMPROVEMENT PROGRAM**

- A. Objective:** Characterize the existing primary process to establish baseline thermal history, chemical changes, and flavor reactions. Identify equipment and process modifications to simplify the primary process to provide a low operating cost and highly flexible primary operation.
- B. Status:** **Thermal Treatment of Burley** - A test plan has been prepared to isolate the response to thermal treatment based on exaggerated drying treatments generated on a lab scale basis. A series of nineteen tests have been defined and will be analyzed chemically, physically, and subjectively. The Sargent tray dryer is being modified and instrumented to provide tight process control for sample generation. The unit will be operational by the end of January.

**Flow Thru Hopper Trials** - A test to establish the quality benefits from cut filler with reduced fines indicated an improvement in firmness occurred with reduced cut filler fines in the cigarette. An estimated 20 mg weight reduction is possible by reducing the less than 1 mm particles by 1.0 percent.

A test is in progress to determine the yield impact (dust generation and removal) of the current cut filler delivery system. A material balance around the cut filler delivery system was conducted in December at the Semiworks using 1500 pounds of Marlboro filler. A similar trial is planned for January with 1500 pounds of a 50/50 blend of Marlboro and DIET to assess the impact on high ET blends.

**Steam Tunnel Prior to Casing** - The 650 for purchase and installation of both the Legg and Sagemuller steam tunnels has been approved. The tunnels have been placed on order with delivery expected in April 1991.

- C. Plans:** Existing information is being compiled to finalize a program outline.

## **II. CLUMP FREE DIET EVALUATION**

- A. Objective:** Confirm previous R&D clump free results and provide recommendation for maximum impregnator fill height to provide clump free benefits. Confirm product benefits at Australia with both Australian and US blend tobaccos.
- B. Status:** The pilot plant trials, initiated to establish the factors contributing to the

reduced product size measured for the clump free process at Australia, have been completed. The major factors identified are:

**Cuts per inch** - the MC DIET feedstock was measured at 26.7 cpi compared to 29.4 cpi measured for Australia. Based on pilot plant results, an improvement of 1.8 units percent 6 mesh could be achieved by decreasing the cuts per inch at Australia to a level equivalent to the MC.

**Drop height** - The drop height of impregnated filler from the Australian impregnator is approximately 10 to 12 feet. A breakage study of impregnated filler demonstrated that increasing the drop height significantly increases product degradation. For reduced cutwidths, as measured at Australia, the effect of drop height on product degradation is even greater.

**Clumped filler** - The MC averages 14.7 percent clumps (by weight) inlet to the tower compared to 7.7 percent at Australia. Analysis of the sieve distribution in clumped versus non-clumped impregnated filler inlet the tower indicated that clumped filler averages 25.0 percent 6 mesh versus 6.4 percent 6 mesh for unclumped filler. This result shows that the unclumped filler is more sensitive to degradation by mechanical equipment downstream from the impregnator, and requires more gentle processing.

The Australian DIET and US blend DIET generated during the testing at Australia have arrived in the US. DIET cigarettes have been produced by the Semiworks. Samples have been submitted for analysis.

- C. **Plans:** All testing for the Australian DIET trials should be completed next month. A summary of the results will be issued in January.

### III. RF TREATMENT OF IMPREGNATED FILLER

- A. **Objective:** Investigate the use of Radio Frequency treatment of impregnated filler to improve the DIET product quality.
- B. **Status:** Radio Frequency drying of impregnated filler will be pursued as a method to improve the DIET product quality. A rental agreement has been signed with Proctor & Swartz, who will supply the RF dryer for evaluation after the unit has been refurbished and fitted with the necessary safety devices.
- C. **Plans:** Evaluation of unit will begin upon delivery and installation expected in mid-January 1991.

### IV. BL PLANT ENVIRONMENTAL STUDY

- A. **Objective:** Identify the point source stack emissions and waste water effluents at the BL Plant. Establish methods for reducing emissions.

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- B. **Status:** Engineering has requested R&D support to verify the BL Plant emissions by conducting a process material balance. R&D has also been requested to address the reduction of exhaust gas volumes at the BL Plant. Point sources at the BL Plant have been identified. The stacks were sampled the week of 11/26/90 under the direction of Engineering.
- C. **Plans:** Material balance sampling around the sheet dryers and stem washing system is scheduled in January and February 1991.

2022201510

PROJECT NUMBER : 1810  
PROJECT TITLE : ART Process Development  
PROJECT LEADER : D. R. Fox  
PERIOD COVERED : December, 1990

## **I. LIQUID ABSORBER PROCESS DEVELOPMENT**

- A. **Objective:** To develop a second-generation supercritical extraction process for nicotine using a liquid absorber rather than stems.
- B. **Results:** Several conceptual designs for a commercial liquid absorber were reviewed with M. W. Kellogg in Houston on December 4 and then presented in Richmond on December 19. During these meetings, it was agreed to recommend a horizontal vessel for the commercial plant. A horizontal design permits lower CO<sub>2</sub> velocities, and thus reduced risk of carryover and foaming, for the same overall vessel size. Kellogg is preparing a more detailed design based on this concept.
- C. **Plans:** The detailed design based on the horizontal vessel will be presented to PM management in January.

## **II. LIQUID ABSORBENT TREATMENT PROCESS DEVELOPMENT**

- A. **Objective:** To develop separation, utilization, and disposal processes for the liquid absorbent effluent from the second-generation ART extraction process.
- B. **Results:** A proposal was accepted from McNamee Advanced Technology for design and consultation services for a pilot biological treatment facility at the pilot plant. Basic design parameters have been discussed, and a preliminary design for the aerobic system has been received. McNamee is now working on additional details as well as the design of the clarifier. The plug-flow basin is about 300 gallons, and the clarifier is expected to be about 100 gallons. Both vessels will be fabricated locally.

McNamee continued to express interest in an anaerobic pretreatment process. They have a small pilot system consisting of a five gallon anaerobic fermentor and a fifteen gallon aerobic vessel and separator available for preliminary testing. They will be loaning this system to us for installation at the pilot plant as well.

Flavor Development has begun separation processing of the Stage 1 effluent from the last series of countercurrent staging runs. Addback testing of either the Flavor Development or Chemical Research recovered fractions will be based on results of analysis by Flavor Development.

- C. **Plans:** Continue process development of the separation, recycle, and disposal processes.

**PROJECT NUMBER :** 1811  
**PROJECT TITLE :** Process Chemistry Development  
**PROJECT LEADER :** G. D. Keritsis  
**PERIOD COVERED :** December, 1990

## **I. CAST LEAF DEVELOPMENT**

- A. Objective:** To investigate appropriate binder systems to be used as an alternative to the current sheet processes.
- B. Results:** The formulations that produced acceptable handsheets with 8-10 percent guar gum and the BL or the BL/RL tobacco feedstocks did not process well in the Cast Leaf Laboratory. The difference in processability was found to be due to the mode of drying of cast sheets in the two laboratories.

While the Cast Leaf Laboratory personnel are working to improve the Glenro dryer to facilitate the processability of these formulations, the Process Chemistry personnel are concentrating their efforts to develop formulations that are less sensitive to drying techniques.

To date, it has been found that the BL/RL tobacco feedstock produces excellent handsheets with 4 percent guar and 6 percent high M.W. pectin when dried on a steam bath, and that this formulation, upon adjusting its pH to 4.25 with 5 percent citric acid, processed very well when dried on the steam box or in the circulating hot air oven at 155°C.

Handsheets of US and Spanish tobacco feedstocks with crossed-over solubles and 4 percent guar were made. The handsheets made with US tobacco feedstock pulp and Spanish tobacco feedstock solubles were found to be acceptable, whereas the handsheets made with the Spanish tobacco feedstock pulp and US tobacco solubles were found to be inferior. The studies in this area are being continued to identify the "binder" in the Spanish tobacco feedstock and the major feedstock component contributing the indigenous water soluble binder.

Laboratory studies indicate that the production of a "HYBRID" RCB/RLB/RLTC sheet to be feasible. Acceptable handsheets were made with the BL/RL tobacco feedstock, 3.75 percent DAP, 7.5 percent  $\text{NH}_4\text{OH}$ , and 5 percent guar gum.

### **C. Plans:**

1. Evaluate the processability of the guar/pectin/citric acid binder formulation at the Cast Leaf Laboratory; Optimize the binder additive levels and produce pound quantities for physical and subjective testings.

2. Optimize DAP/ $\text{NH}_4\text{OH}$ /guar gum levels for the production of an acceptable RCB/RLB/RLTC "HYBRID" sheet at the Cast Leaf Laboratory for physical and subjective testings.
3. Continue the investigations with Spanish tobacco feedstock components to identify major contributors of indigenous water extractable binder in slurry.
4. Continue the characterization studies of other binder systems that could produce acceptable tobacco sheets.

## II. MENTHOLATED RECON

- A. **Objective:** Investigate the feasibility of incorporating encapsulated menthol in reconstituted tobacco sheet.
- B. **Results:** Flavor Development has cigarettes made with 20 percent "mentholated" reconstituted tobacco, and submitted cigarettes for attachment of LARK filters. Cigarettes were mishandled and discarded. The analysis of the recons by Flavor Development for menthol has shown that only 1/3 of the encapsulated menthol survived the sheet making operation.
- C. **Plans:** Remake recon sheets with encapsulated menthol at milder processing conditions and complete the study with machine made cigarettes.

2022201513



PROJECT NUMBER : 1812  
PROJECT TITLE : New Expanded Tobacco  
PROJECT LEADER : E. B. Fischer  
PERIOD COVERED : December, 1990

## **I. BATCH GASEOUS CO<sub>2</sub> IMPREGNATION**

- A. **Objective:** Define process parameters for a batch gaseous CO<sub>2</sub> impregnation process.
- B. **Results:** "Shake-down" runs on the 8-inch tower/impregnator system were initiated using gaseous CO<sub>2</sub> at 800 psig. Modifications to the conveyor transfer device from the impregnator to the tower were identified to improve the handling of the gaseous CO<sub>2</sub> impregnated filler. Installation of a new transfer pump system is scheduled for this week. Design of a larger capacity vaporizer has been finalized; fabrication is being worked out with the vendor. Both modifications are needed to increase the capacity of the pilot system.
- Discussions with PM Engineering and Kellogg were held to come up with a preliminary cost estimate for installing a batch gaseous CO<sub>2</sub> process with a design pressure of 1000 psi in Cabarrus.
- C. **Plans:** Complete modifications on the 8-inch tower/impregnator pilot plant unit and begin experimental grid for evaluating the gaseous CO<sub>2</sub> batch process.

## **II. CONTINUOUS IMPREGNATION PROCESS**

- A. **Objective:** Develop a continuous impregnation process to improve the subjectives of expanded tobacco while maintaining equivalent cigarette filling power to the existing process.
- B. **Results:** Securamax has completed 90% of the design work on the high pressure rotary valve. They analyzed the thermal profile of the valve for heat generation during rotation. The analysis indicated that it is feasible to cool the valve by external cooling providing 85% of the heat is transferred to the housing.

Securamax submitted a quotation to fabricate a rotary valve of pilot plant size (500 lbs/hr) for \$310,783.

A scope document has been generated for the continuous conveyor impregnation process to analyze gas recovery system requirements.

- C. **Plans:** Stress analysis being performed by Securamax will be completed and Securamax will make the necessary drawing modifications to complete the high pressure rotary valve design.

Develop and analyze the gas recovery system for the continuous conveyor impregnation system and detail the design of the belt conveyor.

- B. **Results:** In order to achieve plug flow separation of tobacco, several baffle designs were installed and tested on the 8-inch tower tangential separator. At the Cabarrus flow condition (145 ft/sec), the desired plug flow separation of tobacco from the tower gas was not achieved. However, at significantly reduced flow conditions (30 ft/sec or 20% of Cabarrus flow), acceptable plug flow separation was achieved with two different baffle arrangements. The impact of this finding on the new tower design is being assessed.

A positive displacement feed valve, designed to minimize any pre-expansion of impregnated tobacco, was tested on the 8-inch tower. Preliminary data indicated that the CV behavior was in line with the colder tower temperatures and higher exit tower OV's obtained. Valve design modifications needed to achieve choke-free operation at subzero temperatures have been identified and a redesign of the feed valve is underway.

Based on successful feasibility of the U-tube separator/educto system on the 3-inch tower, scale-up design effort on the 8-inch tower has been initiated.

- C. **Plans:** Complete plug flow testing on the tangential separator. Finalize design modifications to the feed valve. Finalize design of U-tube separator/educto/tangential separator system for the 8-inch tower.

## VII. CHEMICAL STIFFENING

- A. **Objective:** Define a process to chemically stiffen expanded tobacco which will reduce thermal treatment and the associated subjective degradation while maintaining cigarette filling power equivalent to the current process.
- B. **Results:** Extensive testing both in the 3-inch and 8-inch pilot processes has shown that cut filler can be chemically stiffened to enhance product CV. In addition, this stiffening effect continues to be present at much higher than normal tower exit OV's (8-12%) which should make it possible to substantially reduce the thermal treatment and subjective degradation associated with the expansion process. Testing to date has consisted primarily of applying a solution of calcium acetate to uncased DIET feedstock prior to impregnation. A 2.7% add on of calcium acetate with a three hour bulk time produces a product CV increase of about 1.5 cc/g at equivalent tower conditions. Testing is in progress to determine the net result in cigarette performance including subjective evaluation.

Magnesium acetate was evaluated as an alternative stiffening additive because it offered increased solubility over calcium acetate and thus more process flexibility. The magnesium acetate treated filler produced expanded product with improved CV over the control and similar to calcium acetate treated filler. However, its presence increased the equilibrated OV of the product by 1% making it difficult to fully evaluate.

- C. **Plans:** Tests are being planned to evaluate the addition of calcium acetate on strip prior to cutting.

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### III. ALTERNATE CONTINUOUS IMPREGNATION PROCESS

- A. **Objective:** Evaluate the linear pocket processor (LPP) for continuous impregnation of tobacco filler.
- B. **Results:** The design of the model linear pocket processor was completed. The fabrication of component parts is nearly complete and assembly has begun. the unit should be available for testing by mid January.
- C. **Plans:** The model LPP will be used to evaluate the tobacco feed and discharge designs for the prototype LPP.

### IV. TOBACCO COOLING

- A. **Objective:** Develop a continuous cooling process in support of the gaseous carbon dioxide impregnation program.
- B. **Results:** The continuous cooling unit and the associated refrigeration package have been received and are being installed.
- C. **Plans:** Complete the installation of the Frigoscandia test unit in D pilot plant.

### V. ALTERNATE PUFF/DRY/SET PROCESSES

- A. **Objective:** Define alternate means of puffing, drying, setting and reordering impregnated tobacco to improve product subjectives and physical characteristics relative to the present DIET process.
- B. **Results:** A 3-inch development tower arrangement was used to evaluate expansion at elevated temperatures (up to 900°F), temperature quenching of product in line to 150°F, and the U-tube separator concept. Filling power data for product from the test arrangement was similar to that from the conventional 3-inch system. Flavor Development is evaluating the subjective character of the ET produced. The U-tube separator and eductor system operated well, although some breakage occurred in this small scale arrangement.
- C. **Plans:** Conduct subjective evaluation of the high temperature, quick cooled ET. Complete evaluation of physical data. Scale-up U-tube/eductor system for testing on the 8-inch tower.

### VI. TOWER HEAT TRANSFER RATES

- A. **Objective:** Develop process information to define heat transfer parameters for the design of a new expansion tower. Test process concepts on the 8-inch tower leading to process and product improvement.

2022201515

PROJECT NUMBER : 1101  
PROJECT TITLE : Entomological Research  
PROJECT LEADER : D. L. Faustini  
PERIOD COVERED : December, 1990

## **I. CIGARETTE BEETLE (CB) CONTROL PROGRAM**

- A. **Objectives:** (1) To determine if phosphine resistance is occurring in the feral CB, (2) to determine if physiological differences are present in laboratory and field colonies regarding methoprene, and (3) investigate alternatives to conventional tobacco pesticides.
- B. **Results:** CB viability was studied from a diet of ART cut filler. Emergence rates from the ART filler are being compared to standard diet of ground flue-cured tobacco (1). Evaluation of S-methoprene versus R, S-methoprene treated tobacco for CB control has been completed. S-methoprene appears to be twice as active as the R, S-racemic at equal concentrations of the test agents (2).
- C. **Plans:** Complete counting of  $F_1$  emergence. Examine if any relationship exists between CB culture viability and type of processing of the ART filler. Rough draft of S-methoprene report is in preparation.
- D. **References:**

1. Minor, M. F. Notebook No. 9024, pp. 1-2, 9-10.
2. Lehman, R. M. Notebook No. 8740, pp. 87-89.

## **II. SERVICE TO OTHERS**

- A. **Objective:** Provide technical services to areas outside R&D.
- B. **Results:** A insect related customer complaint sample was investigated at the request of Product Audit personnel and results reports (1). An insect infestation involving PM80 cases was reported at the MZM processing plant. Booklice were found to be contaminating the outside of the cardboard cases, but not the contents (2).
- C. **Plans:** Continue to provide support as requested.
- D. **References:**

1. Lehman, R. M. Notebook No. 9014, p. 16.
2. Minor, M. F. Memo to J. R. Thomas, Jr. Insect Infestation of PM80 Cases. November 30, 1990.

PROJECT NUMBER : 1720  
PROJECT TITLE : Analytical Microscopy  
PROJECT LEADER : V. L. Baliga  
PERIOD COVERED : December, 1990

**I. REDUCED SIDESTREAM/FILTRATION/PAPER TECHNOLOGY (Thompson, Sanders)**

A. **Objective:** Examine the ultrastructure of selected cigarette papers, paper additives, and filtration materials in support of the paper technology project.

B. **Results:**

**Papers:** Two 10-028 papers were examined, one from Kimberly Clark and one from Ecusta, and both were found to contain a small amount of wood fibers among the flax fibers. The wood fibers were identified by the presence of pits along the length of the fiber.<sup>1</sup>

**Powders:** Five samples, A through E, of magnesium carbonates that were prepared from aqueous sol gel were examined. Sample A was water washed one time while samples B through E were water washed 3, 5, 7, and 9 times respectively. The morphologies were similar for all samples but the level of K and Cl dropped dramatically after the first washing and disappeared after the third washing.<sup>2</sup>

C. **References:**

1. Thompson, L, "Electron Microscope Analysis of Two 10-028 Papers," Memo to E.B. Sanders, November 15, 1990.
2. Sanders, K., "Examination of Particle Samples 7619-193-A through E," Memo to A. Kallianos, November 26, 1990.

**II. NEW EXPANSION PROCESSES (Sanders, Watson, Baliga, Miser)**

A. **Objective:** Provide analytical support to New Product Expansion.

B. **Results:** Four samples of expanded tobaccos were examined. No conclusions could be drawn from the cross-sectional measurements that were collected and the photographs that were obtained.<sup>1</sup> The cross sectional thickness measurements were only slightly in agreement with the SV measurements. Other methods were discussed to evaluate expanded tobaccos.<sup>2</sup>

A list of commercial facilities and universities that provide high resolution electron microscopy services was compiled. The majority of facilities was comparable in price at about \$2,000 per sample. These facilities are capable of providing high resolution imaging beyond the capabilities of our instrumentation (beyond 0.7 nanometers).<sup>3</sup>

**C. References:**

1. Sanders, K., "Comparison of Expanded Tobaccos," Memo to S. Ganeriwala, December 10, 1990.
2. Watson, D., "Comparison of Expanded Tobaccos," Memo to S. Ganeriwala, December 13, 1990.
3. Baliga, V., and Miser, D., "List of Companies that Provide Microscopy Services," Memo to D. Watson, December 11, 1990.

2022201518

PROJECT NUMBER : 1752  
PROJECT TITLE : Molecular Structure Determination and Materials Evaluation  
PROJECT LEADER : G. Vilcins  
PERIOD COVERED : December, 1990

#### I. NMR ANALYSIS OF FLAVORS

- A. Objective: To use NMR to determine the structure and purity of novel flavor release compounds.
- B. Results: Several compounds in a series of alkyl pyrazines and pyridines were analyzed by  $^1\text{H}$  NMR. NMR was used to determine the positional isomers and confirm the overall structures. Two isomers of cyclopropylpyrazine and cyclopropylpyridine were analyzed by NMR. Several menthyl carbonates of methyl- $\alpha$ -D-glucopyranoside were analyzed by  $^1\text{H}$  and  $^{13}\text{C}$  NMR in order to determine the structure and purity.
- C. Conclusions: NMR is the method of choice for these types of analyses. The overall purity of these compounds were better than 95% in most cases.
- D. Plans: NMR will continue to support to the flavor-release and pyrolysis programs.
- E. Reference:

Bassfield, R., PM Notebook #7398, p. 176.

#### II. $^{13}\text{C}$ CPMAS NMR OF CALCIUM HYDROXIDE TREATED EXPANDED TOBACCO

- A. Objective: Determine the degree of esterification of pectin isolated from expanded tobacco and the effect of calcium hydroxide treatment on expanded tobacco and the isolated tobacco pectin.
- B. Results: The degree of methyl esterification of the pectin in expanded tobacco was found not to be significantly affected by calcium hydroxide treatment. Calcium ion binding to pectin carboxyl groups was indicated by a downfield shift of some of the carboxyl resonances.
- C. Conclusions: Esterification of tobacco pectin is not effected by calcium hydroxide under the current treatment regime. Interaction of free pectin carboxyl groups does occur and may influence tobacco expansion.
- D. Reference:

Wooten, J., " $^{13}\text{C}$  CPMAS NMR of Calcium Hydroxide Treated Expanded Tobacco and Isolated Pectin," Memo to W. Hempfling and K. Shafer, November 27, 1990.

### III. MS ANALYSIS OF INORGANIC MAGNESIUM COMPOUNDS

- A. Objective: To explore the feasibility of in situ analysis of inorganic magnesium compounds by tandem mass spectrometry.
- B. Results: Fast atom bombardment and direct probe low voltage EI ionization using the JEOL SX 102/ SX 102 tandem mass spectrometer have been explored as methods for formation of characteristic ions from inorganic magnesium compounds. Fast atom bombardment ionization yielded some potentially informative ions.
- C. Conclusions: Experiments still are in progress.
- D. Plans: The potentially informative ions will be investigated by MS/MS. If this work yields satisfactory results, analysis of these compounds on paper will be pursued.
- E. Reference:  
Jensen, N., PM Notebook #8910, p. 80.

2022201520



**PROJECT NUMBER :** 1757  
**PROJECT TITLE :** Analytical Flavor Specifications  
**PROJECT LEADER :** M. L. Zimmermann  
**PERIOD COVERED :** December, 1990

**FLAVOR SPECIFICATIONS AND CERTIFICATION**

- A. **Objective:** To develop analytical and sensory specifications for current, incoming flavor materials and to transmit specifications and methods for monitoring specifications to appropriate groups. To certify that PMI export flavor materials meet GFO, to issue a certification of analysis and to transfer methodology and certification to the Flavor Center.

B. **Results:**

A third off-site visit to one of the flavor vendors has resulted in favorable acceptance of the proposed specifications for their products. Exchange samples both simple and complex are planned and these are to be handled by the Flavor Center. Communications with other vendors as to the methodology used to specify the flavors has been examined and communications with key individuals is in progress.

A set of 168 DM codes are in the final stages of the specification process. A series of vendor packages are now being prepared for release to these vendors. A series of materials still requires development of methods suitable for use in the QA environment for products not amenable to our current procedures.

Material was harvested from the greenhouse and initial attempts to isolate a marker compound have met with success; however, the identity of this compound has not been determined. Attempts by Chemical Research personnel to isolate marker components for the other botanical material has also met with success. Efforts are in place to develop suitable approaches for inclusion of these procedures in the certification program.

A problem associated with one of the PME materials was investigated at the request of M. Murray. Subsequent sample exchanges and component analyses determined that a treatment procedure in the method for analysis at PME was hydrolyzing the material from certain components within the blend.

- C. **Plans:** Continue the certification of the PMI samples, continue to effect a smooth transition of the procedures used for this process to the Flavor Center, issue the FRG Direct Materials, complete the specification process for the vendors on hand and continue the visits with key vendors on technical issues.

PROJECT NUMBER : 1902  
PROJECT TITLE : Cell/Tissue Culture Research  
PROJECT LEADER : D. M. Teng  
WRITTEN BY : D. K. Chadick  
PERIOD COVERED : December, 1990

## **I. TOBACCO MICROBIOLOGY**

**A. Objective:** To develop methods and evaluate the microflora in tobacco materials.

**B. Results:**

1. 1989 Bright Mold Evaluation Study

The sampling and analyses of samples from the 1989 bright crop was completed. The microbial populations were evaluated from a total of 195 samples of bright tobacco. These results are being analyzed.

2. 1990 Bright Study

Sampling and microbial analyses (bacteria, mold, and yeast) were continuing from samples of the 1990 bright crop. To date, a total of 345 samples have been evaluated. There are no final results to report at this time.

3. Bactometer® Study

The data collection and statistical analyses have been completed for the comparison of plate-count numbers vs. Bactometer® counts. Based on the statistical analyses, it was determined that the Bactometer® was not a suitable alternative for the current plate-count procedure (1).

4. Burley/Bright 4°C Storage Study

Samples of burley and bright tobaccos were stored for 28 days at 4°C. The microbial populations were evaluated at time zero and after 1, 2, 3 and 4 weeks of storage. The final results showed no significant change in the microbial populations regardless of storage time (2,3). Therefore, burley and bright tobaccos can be stored up to 28 days at 4°C, if necessary, prior to microbial analyses.

**C. Plans:** (1) Continue data analyses and document final results, (2) continue sampling and microbial evaluations for the 1990 bright crop and conduct a similar study with the 1990 burley crop, (3) prepare a completion report/memo with the final results.

1902-2

**D. References:**

1. Jones, R. M. Comparison of Bacterial Count Methods. Memo to D. Chadick; 1990 December 6.
2. Chadick, D. Notebook No. 8825 p. 197.
3. Gaines, O. Notebook No. 8904, pp. 162-164, 172, and 175.

2022201523

PROJECT NUMBER : 1904  
PROJECT TITLE : Tobacco Physiology and Biochemistry  
PROJECT LEADER : D. J. Ayers  
WRITTEN BY : S. Wahab  
PERIOD COVERED : December, 1990

## **I. LOW NICOTINE STUDY**

- A. **Objective:** To investigate the biochemistry of the nicotine biosynthetic pathway at the putrescine N-methyltransferase (PMT) step and specifically to isolate PMT from tobacco root extracts.
- B. **Results:** The roots from Group 27 hydroponically grown, Burley 21 tobacco plants were harvested. These plants were very small in height and leaf size. The average root weight in this group of plants was also low compared to previous groups. Batches from these roots are being processed to determine PMT specific activity in this harvest (1,2,3).

Preparation of antibodies against PMT protein was initiated by isolating the 60 kD protein band from SDS-PAGE gels. PMT protein bands were sent to a contract laboratory for antibody production (4,5).

Several DEAE/AHS columns were used to process previously prepared phenyl-Sepharose fractions containing PMT enzymes. Good enzyme activity was obtained from some of these fractions (1,2,3,4). In addition, several previously prepared phenyl-Sepharose PMT samples were tested for protease activity. Two different temperature experiments (37°C & 4°C) were performed to assay for protease activity in these samples along with protein control. None of the Phenyl-Sepharose samples showed any protease activity (3).

A protein of interest to Miller Brewing (MP100) and bovine serum albumin (BSA) were successfully electroeluted from SDS-PAGE in sufficient quantities for cyanogen bromide (CNBr) digestion. Digestion of the purified proteins with CNBr showed distinct peptide fragments. The digestion of the partially purified MP100 extract revealed the same pattern of CNBr generated peptides as the SDS-PAGE purified MP100 (6). A PMT sample (4) was used for preliminary CNBr digestion. PMT was electrotransferred to an Immobilon-P filter and then digested with CNBr while still attached to the Immobilon-P. Four peptide fragments were visualized but at very low concentrations.

New preparations of poly(A<sup>+</sup>) RNA from mature tobacco roots were run on an agarose-formaldehyde gel. Northern blots were prepared by blotting the RNA from the agarose-formaldehyde gels onto nitrocellulose membranes (S&S). Fragments of approximately 700 and 400 bp, isolated from tobacco overexpressed clone pR17, were radioactively labeled and used as DNA probes to hybridize to total and poly(A<sup>+</sup>) RNA northern blots. A distinct band of more than 1,000 bp long was visualized due to hybridization of these fragments to total and poly(A<sup>+</sup>) RNA. This indicates that poly(A<sup>+</sup>) preparations are suitable for oligonucleotide hybridization.

studies. Radioactive labeling of five oligonucleotides, derived from the putative PMT protein sequence, was initiated (7).

Several pGEM DNA standards were run on the AB1 automated DNA Sequencer to establish conditions for using Bio-Spin columns. In addition, DNA was prepared from several tobacco root clones to be used for sequencing (7,8).

Amplification of the putative PMT gene was attempted using three putative PMT primers and oligo dT via the polymerase chain reaction (PCR) method. A cDNA first strand was prepared from total and poly(A+) RNA. Amplification of this first cDNA strand has also been attempted by PCR (9).

- C. Plans: Continue to prepare partially purified PMT from phenyl-Sepharose fractions through DEAE/AHS columns for CNBr digestion. Continue to process the newly harvested tobacco roots. Continue to develop a high radioactive labeling method for putative PMT synthetic oligonucleotides and start northern blot hybridization with these oligos. Attempt to amplify PMT sequences using PCR technology and clone a tobacco root cDNA fragment into a plant vector. Attempt to sequence several tobacco root clones.

D. References:

1. Lyle, J. Notebook No. 8856, p. 193.
2. Turner, D. Notebook No. 8973, p. 165.
3. Davies, S. Notebook No. 8976, p. 159.
4. Nakatani, H. Notebook No. 8384, p. 162.
5. Yu, T. Notebook No. 9002, p. 61.
6. Bower, P. Notebook No. 9032.
7. Wahab, S. Notebook No. 8983, pp. 139-141.
8. Michalik, T. Notebook No. 9036, p. 25.
9. Malik, V. Notebook No. 8974, p. 64.

2022201525

PROJECT NUMBER : 2500  
PROJECT TITLE : Fundamental Chemistry  
PROJECT LEADER : J. I. Seeman  
PREPARED BY : J. A. Fournier  
PERIOD COVERED : December, 1990

## **I. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES**

(Fournier, Kallianos, Paine, Podraza, Secor, Seeman)

**A. Objective:** To develop inorganic materials for novel applications for reduced sidestream, for burn-rate modification, and enhanced subjectives in cigarettes and for required properties in novel smoking materials.

**B. Results and Plans:** RSA Corporation's laboratory evaluation of the preparation of magnesite via a hydrothermal reaction of  $\text{Mg}(\text{OH})_2$  with  $\text{CO}_2$  has been completed. Samples, all reported to be greater than 95% conversion, have been received and are in the process of being evaluated.

Subjective results on two cigarettes, one containing a paper filler of "recrystallized"  $\text{Mg}(\text{OH})_2$  and the other containing Reheis  $\text{Mg}(\text{OH})_2$  powder, were received. Both cigarettes which, gave 80% sidestream smoke visibility reduction with static burn times of approximately eleven minutes, exhibited positive subjectives. Microscopy results to determine the morphology of the two samples are pending and requests have been made for additional papers and cigarettes.

Smoking data on two sets of cigarettes prepared with papers containing mechanical mixtures at 92% to 8% and 78% to 22% of Baymag C magnesite and freshly prepared MgO, respectively, were received. Sidestream reductions of 52% for the paper containing a higher percentage of Baymag C magnesite and 67% for the paper containing a higher percentage of MgO with static burn times of 8.4 minutes and 7.6 minutes, respectively, were exhibited. It was originally hoped that the addition of MgO to magnesite would help improved ash integrity, however, this was not observed. Subjective evaluations are pending.

To support patent application PM-1511, requests for additional handsheets containing magnesite,  $\text{Mg}(\text{OH})_2$ , and mixtures thereof have been made.

The kinetics of the dissolution of eitelite in water was investigated. The rate of solution of sodium decreased significantly with time, as the concentration of sodium in the solution increased. The rate of hydrolysis was found to be significantly slower in dilute sodium carbonate (1% total sodium). Thus it appears that low values of sodium can significantly retard the decomposition of eitelite, and improve the chances for papermaking.

The reaction between calcium hydroxide (portlandite) and magnesium bicarbonate was repeated at the same four ratios as conducted previously, so as to isolate the product as a slurry. Several of the previous preparations had proved difficult to resuspend in water after drying. X-ray results for the earlier set of reaction products

show calcium carbonate to be present as both calcite and aragonite. At low ratios of portlandite to magnesium, the magnesium phases are nesquehonite and/or hydromagnesite. At high ratios, the magnesium phases are hydromagnesite and/or brucite. Residual portlandite was also detected, probably occluded in the larger particles. In the latest preparations, now submitted for analysis, and awaiting results, coarse particles were removed by decantation.

Smoking results have been completed for three sets of cigarettes prepared with papers containing 20% MM  $\text{CaCO}_3$  and 5 to 10% (depending on retention) aqueous "sol-gel" derived hydromagnesite/magnesium hydroxide mixtures with ratios of 50:50, 60:40, and 70:30, respectively. Sidestream reductions of 41% to 52% with static burn times of 7 to 8 minutes were observed with these models. The subjectives of the latter model were judged to be acceptable, exhibiting no negative attributes.

The use of calcium carbonate as a co-filler in "mag carbonate" handsheets provides porosity to the paper, gives shorter drainage times in the handsheet making process, and enhances the ash characteristics of cigarettes prepared from these papers. Sidestream smoke data of cigarettes prepared with papers containing "mag carbonates" and different calcium carbonate co-fillers show differences in visibility reduction. Evaluations of other calcium carbonate co-fillers are being conducted to determine if there are advantages in using one calcium carbonate sample over another.

Reevaluation of several "mag carbonate" samples synthesized earlier in the year have shown the samples to have undergone an aging process, i.e. a change in crystallinity. Work is being conducted to better understand this process and how it might effect the smoking properties of a given material.

Studies are being conducted to better understanding the nature of the soluble magnesium bicarbonate species believed to be the intermediate to solid "mag carbonate" phases. In one study, the reaction, in water, of magnesium hydroxide with carbon 13 labelled carbon dioxide was conducted, and the resulting solution was examined by  $^{13}\text{C}$  NMR. Initial results indicate that the reaction proceeds slowly.  $^{13}\text{C}$  NMR shows only one peak at  $\sim 165$  ppm. Identification of the chemical species giving rise to this signal will require additional standards. The solid material filtered from this reaction is also in the process of being characterized. In another study the ratio of carbon dioxide to the dissolved magnesium (crucial for developing reproducibility in the above chemistry) is being examined using a gravimetric technique. A plot of carbon dioxide content versus pH at constant magnesium content is the ultimate goal of the latter study.

In the interest of comparing the mainstream smoke chemistry of low sidestream cigarettes sized with hexapotassium phytate versus those sized with conventional fluxing agents, cigarettes have been prepared from E2560  $\text{CaCO}_3$  papers sized with either  $\text{K}_6$  phytate or  $\text{K}_2$  succinate. The cigarettes have been submitted to Analytical Research for the requisite analyses.

A series of intermediate basis weight papers containing different levels of  $\text{CaCO}_3$  at different porosities were sized with tripotassium and hexapotassium phytate. At low levels of application the tripotassium salt increased the porosity of the papers substantially and had no effect on the sidestream smoke level of cigarettes made from these papers. At slightly higher levels of application the hexapotassium phytate showed no effect on paper porosity and exhibited a modest level of sidestream reduction with acceptable static burn times. Further evaluations of these materials in sizing applications are planned.

Hexapotassium phytate solutions at 26% and 50% solids have been used to coat bands onto standard cigarette papers via the gravure process. The strips of paper having band-coatings of 7mm wide have been used to prepare machine made cigarettes. These samples have been submitted to Physical Research for evaluation of mass burn-rate as well as other routine analyses.

During the month, we have reviewed proposals to Philip Morris as well as to the Virginia Center for Innovative Technology from Dr. Richard Zallen of Virginia Tech. A response to Dr. Zallen will be prepared after we assess completely the impact of his proposals to PM interests. We have also reviewed and discussed a preliminary proposal for 1991 from Dr. Donald Schleich of N.Y. Polytechnic University. This proposal should be finalized shortly.

## **II. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS** (Howe)

- A. **Objective:** To develop techniques to remove selectively nicotine and other alkaloids from aqueous tobacco processing fluids.
- B. **Results and Plans:** Old samples of MPC solution from water column runs at the BHPP have been discarded. One of the solutions from April of this year was reanalyzed for nicotine concentration at the request of D. Teng as a comparison to the original analysis done in April at the BHPP. No change in the nicotine concentration was noticed even though the sample was darker in color and smelled "degraded" or "fermented".

## **III. MISCELLANEOUS** (Secor)

One of a series of nicotinoids for R. Carchman has been prepared and work continues on the preparation of others.

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2500-4

Structures of Substrates

calcium carbonate, calcite  $[\text{CaCO}_3]$

eitelite  $[\text{Na}_2\text{Mg}(\text{CO}_3)_2]$

hydromagnesite  $[\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \bullet 4\text{H}_2\text{O}]$

magnesium hydroxide, brucite  $[\text{Mg}(\text{OH})_2]$

magnesite  $[\text{MgCO}_3]$

nesquehonite  $[\text{MgCO}_3 \bullet 3\text{H}_2\text{O}]$

phytate = myo-inositolhexaphosphate

2022201529

PROJECT NUMBER : 2501  
PROJECT TITLE : Smoke Chemistry  
PROJECT LEADER : R. A. Comes  
PERIOD COVERED : December, 1990

## **I. SIDESTREAM SMOKE CHAMBER**

A. **Objective:** Operate an environmentally controlled chamber to measure selected components of sidestream smoke.

B. **Results:** Some components for the chamber adsorption/desorption gc/ms organic compound analysis system have been received. Preliminary plans for installation, testing and training are being coordinated with OI Analytical and Hewlett-Packard.

Changes have been made by personnel of the Computer Applications Division to the chamber computer software to facilitate the transfer of additional Factory Link data columns to the chamber database.

Multiple chamber maintenance items have been addressed in anticipation of an increased work load shortly. A water softener system has been installed to attempt to prevent the previous problems with the chamber boiler system.

A memo has been written describing the multiple problems encountered and successful conclusions reached in transferring the nicotine analysis to the chamber laboratory.

Initial checks of the chamber flow system have been made in anticipation of upcoming studies by personnel of the Biochemical Research Division.

C. **Plans:** Chamber runs will continue as required.

## **II. SIDESTREAM SMOKE**

A. **Objective:** Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream semivolatiles and gas phase; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.

B. **Results:** The schlieren system for visualization of cigarette sidestream smoke has again been set up. Video capability for both schlieren and static and dynamic sidestream smoke now exists. The IR camera is being reinstalled. An IR applications course will be taken in January by a project member.

C. **Plans:** CORESTA smokings and other support for the sidestream odor modification program will continue. The schlieren and IR camera systems will be used in support of the sidestream smoke reduction program.

### **III. MISCELLANEOUS**

Pyrolysis gc/ms analyses were conducted on three fungicide samples and on samples with potential utility in the sidestream odor modification program.

In conjunction with personnel of the Biochemical Research Division, initial carbon-14 studies have begun to investigate the bacterial decomposition of nicotine.

2022201531

PROJECT NUMBER: 2520  
PROJECT TITLE : Flavor Research  
PROJECT LEADER : Y. Houminer  
PERIOD COVERED : December, 1990

## I. FLAVOR RELEASE TECHNOLOGY

- A. Objective: To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.
- B. Results: We continue to explore the synthesis and pyrolysis of various menthol release agents. The reaction of di-O-acetyl-L-tartaric anhydride with menthol was carried out at 145°C. After workup, a foamy hygroscopic solid was obtained which was recrystallized from hexane to give a solid with a sharp melting point. A sample has been sent for elemental analysis and NMR.

Several other menthol release agents were obtained. Dimethyl oxalate was synthesized and purified by flash chromatography. A pure sample of the material was obtained and has been submitted for NMR and elemental analysis. Purification of the mono-menthyl diglycolate has been completed. Samples have been submitted for NMR and elemental analysis.

A large sample of beta-menthyl itaconate was purified to give 54gm of pure ester. An additional 10 grams of calcium beta-menthyl itaconate has been prepared for use by Flavor Development in spray application for subjective evaluation.

Dr. L. Harwood from Oxford University has informed us that his group has discovered that they can get about 70% distilled yields of MIC using sodium hydride instead of potassium hydride. They are planning to use the same conditions to prepare isopropenyl chloroformate.

We have reported in the past that phenethyl  $\beta$ -D-glucopyranose reacts with menthyl chloroformate in dioxane to give 66% yield of the 6-O-carbonate. We have recently discovered that methyl  $\alpha$ -D-glucopyranoside reacts with menthyl chloroformate in pyridine to give a mixture of compounds: the 6-O- and 2-O-carbonates, and a third component which is believed to be the 2,6-O-dicarbonate. The difference in selectivity can be attributed to the solvent effect as we have demonstrated before in the reaction of glucose with menthyl chloroformate.

We continue to explore the grafting of the trans-2,3-cyclic carbonate of Aromatek 245 to other carbohydrates using xylital as a model compound. Several experiments were carried out. It was observed that in the presence of triethylamine, the cyclic carbonate group was extremely sensitive to moisture, and the compound decomposed spontaneously to Aromatek-245. In the presence of pyridine, however, the cyclic carbonate group was extremely stable and only very little decomposition was observed. On the other hand, the reactivity of the cyclic carbonate with xylitol was very slow, even in the presence of triethylamine. So far most of the experiments resulted in decomposition of the cyclic carbonate, or recovery of starting material.

We continue to investigate other reaction conditions.

Samples of both CR-2950 and CR-2951 have been delivered to Flavor Development for evaluation. Both compounds are designed to release  $\alpha$ -hexylcinnamaldehyde.

## II. FILTER MATERIALS AND PAPER TECHNOLOGY SUPPORT

- A. **Objective:** To find outside suppliers for large quantities of new filter materials and inorganic paper additives.
- B. **Results:** R.S.A. has completed the laboratory scale development work on the production of magnesite by hydrothermal carbonylation. Both Reheis magnesium hydroxide powder and 30% paste have been converted to magnesite in about 24 hrs. The paste gave 99% and the powder 98% magnesite respectively. Samples will be arriving the week of December 17th and evaluation of the material will be carried out by J. Fournier and B. Rogers.

Based on the above results, R.S.A. feels that they will be able to conduct this chemistry in their large pressure reactor. They will be supplying approximate cost, size, and available production dates later this month. Final production will not be run until the lab samples have been evaluated and the projected cost reviewed.

Work continues at Forest Products Labs (FPL) on the large scale acetylation of cellulose. In a recent run (batch #6) the degree of acetylation was 0.44, which is about half of the target degree of acetylation.

Analytical research has completed the analysis of the papers made from the FPL samples. Results show that the papers are uniformly lower (between 15 to 30%) in acetyl content than the original samples. NMR analysis confirms these results. Bob Rogers is evaluating the paper making process to see if the loss may be there.

Dr. David Giachardi (Director of R&D, Courtaulds) and Dr. David Randall (Director of R&D, Courtaulds Acetate) met with PM scientists in late November. Courtaulds is willing to enter into a cooperative business agreements with PM. The two primary areas will be Fibril systems and partially acetylated cellulose systems. Agreements are being prepared.

## III. FLAVOR CHEMISTRY

- A. **Objective:** To obtain flavors for subjective evaluation and odor profiling. To isolate and identify tobacco components which are sensorially significant.
- B. **Results:** We continue to prepare different flavors which are not commercially available for evaluation in the odor profile program. Isopropenyl pyrazine was prepared from ethylpyrazine to compare its odor character (popcorn-nutty) to the material previously prepared from acetylpyrazine. The odor character was found to be unrelated to the method of preparation, thus indicating the absence of any impurities in the product.

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2-Isopropenylpyridine was prepared from 2-ethylpyridine. Surprisingly its odor character is an intense green-floral, not popcorn nutty.

Free-radical alkylation of pyridine using n-valeric acid gave a number of butylated products. A similar reaction with thiazole gave no significant amount of product and only 15% recovered unreacted thiazole.

2022201534

PROJECT NUMBER : 2526  
PROJECT TITLE : Greenhouse Operations and Cooperative Tobacco Variety  
Evaluation Programs  
PROJECT LEADER : R. T. Bass  
PERIOD COVERED : December, 1990

**I. GREENHOUSE STUDIES AND SUPPORT ACTIVITIES**

(R. Bass, G. Newell and G. West)

A. **Objective:** Maintain the R&D Greenhouses, conduct plant research studies, provide greenhouse-grown tobacco materials for support of other R&D programs, and provide requested assistance for special projects.

B. **Results:** During this period hand fabricated cigarettes were made for samples H1052, H1053, and H1054 as requested. These cigarettes were for the lotus program and are to be utilized in subjective evaluations. The Rizla RYO maker was used to make these cigarettes. A total of 18 Greenhouse grown tobacco samples from the Hydroponic Groups 24, 25, and 26 were ground on the Wily Mill using a 20 mesh screen. These samples will be submitted for total alkaloid analyses.

The air pressure control scrub tanks for the Greenhouse plant growth chamber system contained NaOH solution, which was assayed and then disposed of in the proper manner as directed by the R.S.O. An order has been placed for the fabrication of two new plexiglas tanks to replace the old ones.

The leaves from the eighteen Dryopteris Filix-mas fern plants have been harvested and provided to M. Zimmermann. He was also provided with a quantity of air dried leaves from the bittersweet Solanum Dulcamara plants.

The harvest of the forty Burley 21 plants for Group 27 has been completed and the root tissue provided to Project 1904. Two plants were selected and measurements for leaf size and weight were made. Leaf samples were oven dried for analyses.

The regular Greenhouse plant production activities were completed, including the preparation of 220 gallons of nutrient solution, the harvesting of tobacco and ferns, several insecticide applications, the performance of all culture practices, and the seeding and transplanting of Burley 21, K-326, and Speight G-28 tobaccos.

The regular Greenhouse operational and maintenance tasks have been performed on schedule including the rebuilding of the Kool Cel system water pump in order to have a spare on hand, if needed.

A sample containing 50 fresh green tobacco leaves was provided to Project 6502 as requested for use in their B.O.B. display.

C. **Plans:** Greenhouse operations and maintenance will be continued along with all requested support activities.

**D. References:**

1. G. Newell, N.B. No. 8762
2. G. West, N.B. No. 8559
3. R. Bass, N.B. No. 8999

**II. COOPERATIVE TOBACCO VARIETY EVALUATION PROGRAM**

(R. Bass, G. Newell and G. West)

- A. **Objective:** To participate in the cooperative tobacco industry program in order to assure that the quality of all new tobacco varieties meets or exceeds all requirements as defined by minimum standards program.
- B. **Results:** The annual meeting of the Flue-Cured Tobacco Variety Evaluation Committee was attended in Raleigh, N.C. For the 1990 Regional Farm Test, evaluation of the data for chemical, physical, agronomic, and smoke flavor characteristics showed the following test entries to be acceptable and eligible for release as new varieties in 1991. They are: NC7029 USDA, Speight G-117, NC6085 USDA, RG8, Reams 137, and RG22. The entry sponsors indicated that Speight G-117 will be released as Speight G-117 and that Reams 137 will be released as Reams 713. Entry Speight G-122 was unacceptable because of genetic instability, and entry Reams 124 was unacceptable because of undesirable smoke flavor. The control varieties for the test were NC 2326 and NC 95. For the 1990 Regional Small Plot Test, there were a total of 24 entries, 15 of which were acceptable and are eligible for advancement to the 1991 Regional Farm Test. Following the regular meeting, a business meeting session was held for consideration and discussion of several suggestions that have been offered regarding the organization and implementation of the Regional Program.

The analyses for the 399 samples from the 1990 South Carolina Mammoth Tobacco Test have been completed. The total alkaloid and sugar results have been reported to personnel at the Pee Dee Research and Education Center.

On December 4th the Annual Tobacco Day Meeting, presented by the N.C.S.U. Plant Pathology Extension Service, was attended at Raleigh, N.C. Their crop information showed the 1990 N.C. tobacco crop disease losses (6%) to be ranked in the following order: 1.) Black Shank, 2.) Granville Wilt, 3.) Mosaic, 4.) Root Knot, 5.) Brown Spot, and 6.) Target Spot. The crop survey showed the 1990 flue-cured tobacco variety planted acreage to rank as follows: 1.) K-326, 2.) K-399, 3.) NC 37 NF, 4.) K-358, 5.) Speight G-28, and 6.) K-149. There is some concern about the Tobacco Spotted Wilt Virus disease because there are no varieties available which are resistant to it. The only effective control practice presently available is to use disease free transplants.

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The combined 1990 Burley Tobacco Variety Evaluation Test including tobaccos from North Carolina, Virginia, Tennessee, Kentucky, and Ohio, was attended in Greeneville, TN, at the Tobacco Experiment Station. In cooperation with the PM Leaf Department, a total of 635 leaf tobacco samples from the Regional Farm Test, the Regional Small Plot Test, and the Regional Growth Control Regulator Test were inspected. The tobaccos were evaluated for color, body, texture, and quality suitability for PM use.

C. Plans: To continue to participate in the Cooperative Tobacco Variety Evaluation Program.

D. References:

1. R. Bass, N.B. No. 8999
2. G. Newell, N.B. No. 8762
3. G. West, N.B. No. 8559

2022201537

PROJECT NUMBER : 2705  
PROJECT TITLE : Tomorrow  
PROJECT LEADER : R. W. Dwyer  
PERIOD COVERED : December, 1990

#### **I. PROJECT TOMORROW**

A. **Objective:** Explore the feasibility of controlling the burn rates and puff counts of cigarettes through filler and wrapper modifications.

B. **Status:** One means of reducing cigarette puff count is by reducing the weight of tobacco in the rod. In an effort to quantify the weight reduction which small-lamina material affords, we had cigarettes fabricated from nine different blends of fillers. The base filler was a conventional blend without expanded tobacco or stem. To that we added BBOSL at four levels with a maximum of 32 percent by weight. For comparison, we used the same base filler with varying levels of DIET tobacco. The cigarettes from these blends were made to a common firmness and smoked for puff count and delivery. The results show that BOSL can be used as a direct one-to-one replacement for DIET tobacco, at least up to the 32% level. The weights at firmness, puff counts, and tar deliveries were the same for both the BBOSL and DIET blends at the same addition level.

Additionally, we examined the weight/firmness relationships of four expanded tobaccos. Cigarettes were made with DETA, XTK, BLDET, or BBET as the only filler component. These samples were made to the same firmness, and the BBET cigarettes had significantly lower tobacco weights and puff counts than the other materials. The results suggest that the more burley tobacco in the expanded blend, the greater the weight reduction.

C. **Plans:** At this point, we have found fillers which can be used to reduce the weight of tobacco in cigarettes. We have previously reported the results of studies which show that the type and amount of additives in wrappers can alter the amount of tobacco burned during a puff. Based on the results of these two studies, we plan to design cigarettes at reduced mass burn rates which will provide conventional deliveries and puff counts.

PROJECT NUMBER : 2706  
PROJECT TITLE : New Expanded Tobacco  
PROJECT LEADER : T. M. Howell  
PERIOD COVERED : December, 1990

## I. EXPANDED TOBACCO

A. **Objective:** Determine if the CV of expanded filler can be improved and/or if post expansion collapse can be reduced by pretreating DIET feed with approved additives.

B. **Results:** Last month it was reported that pretreating DIET filler with calcium hydroxide and bulking for 24 hours increases the CV of the expanded filler by about 1.8 CV units. Experiments this month have confirmed that work. It was further observed that 1%, 3% and 5% addition levels of CaOH gave almost equivalent results. Calcium acetate and a combination of calcium acetate plus KOH were shown to produce CV improvements similar to CaOH in the expanded filler. Pilot plant trials were scheduled and executed to optimize the use of these additives. The results of those tests are pending and will be reported separately.

An increase of about one CV unit for bright filler was achieved with the addition of 5% CaOH and 24 hour bulking at 30% OV. This stiffening improvement was obtained without subjecting the tobacco to the expansion process. In comparison, a 0.6 CV unit gain was made possible by bulking the filler at 30% OV for 24 hours without any additives. Preliminary trials with calcium acetate have shown promise but in all cases the extent of increase is dependent on the amount of additive added, bulking moisture and bulking time.

C. **Conclusions:** Allowing tobacco to stand for an extended period of time at high OV's is not practical under the current tobacco processing scheme. If CV gains can be attained at shorter bulking times, lower OVs and without a change in the tobacco subjective character, then it may be possible to improve the filling power of the total blend.

D. **Plans:** Additional studies are now underway to investigate the effect of alternate additives on each of the blend components and to define the minimum treatments required to yield optimum improvements. Contacts have also been made within the Analytical Division to initiate studies that will lead to an understanding of the mechanisms involved and with Manufacturing Primary to determine at what points during processing application may most easily be implemented.

## II. EXPANDED TOBACCO

A. **Objective:** Determine if post expansion collapse can be minimized by staging the heating process during expansion.

B. **Results:** DIET filler impregnated with liquid CO<sub>2</sub> was rapidly heated by passing 6000F air through the filler at a high velocity for about 2 seconds. The heated filler

was then transferred to a second heating zone where 225° was passed through for about 10 to 12 seconds. Immediately after the second stage heating the tobacco was quenched in liquid nitrogen. SVs were measured on the filler before and after equilibration at 60% RH in an environmental chamber. A series of three tests using the above procedure was completed with no improvement shown in SV over the normal DIET expansion process.

- C. **Conclusion:** The above procedure was designed to give a quick indication of the feasibility of staged heating and wasn't well controlled; however, the results do not present any worthwhile evidence for pursuing this expansion scheme further in the laboratory.
- D. **Plans:** No further work is planned on this objective. Continue efforts to decrease the rate of expansion as described last month using the modified bench top expansion unit. Determine the precise temperature at which the impregnated CO<sub>2</sub> is released and expansion occurs.

2022201540

PROJECT NUMBER : 2707  
PROJECT TITLE : Vision Inspection Technologies  
PROJECT LEADER : R. J. Maher  
PERIOD COVERED : December, 1990

## I. PACK INSPECTION SYSTEM

A. Objective: Develop and implement an on-line cigarette pack inspection system with the capability for global inspection.

B. Results: The new pack gauge blocks were received from the engraver. These new blocks differ in that the anodization was a gray instead of black. This gives sufficient color contrast between the engraved lines and the block as well as the block and the background.

Laboratory testing of the OSIRIS pack inspection system has been initiated and its inspection functionality has been demonstrated. Several serious Matrox software bugs have been encountered which delayed completion of the system software. One of the problems limits the system's capability to simultaneously inspect packs and present graphical information. We are working closely with Matrox to resolve this situation to avoid impacting system performance during the factory test.

C. Plans: Resolve the Matrox software bugs and complete the software development. Begin extensive laboratory testing of the prototype. Add the low variance modifications to the training and inspection algorithms.

## II. PRINT WEB INSPECTION

A. Objective: Develop a system for the global inspection of print web on the printing press.

B. Results: An optics table was installed with a motorized drum to simulate a print web press. ETI installed its modified Dalsa Camera. Since the fast clocking ETI control board wasn't ready, a board that can accommodate a surface speed of 200 ft/min was used. This permits us to obtain image data with the ETI frame enable and encoder techniques. ETI will have the fast clocking camera (800 ft/min surface speed) ready before January and will substitute it for our present system. Several camera interface and imaging board problems were encountered and were adequately solved so that we can obtain monitor images of a sample web surface. The monitor images are very good in comparison with either the standard Dalsa images or with standard RS170 images.

The computer software that has the capability to save acquired images was installed. Some problems exist with the software and are being addressed.

The interfacing of a line scan camera to the Trapix system is being pursued.

- C. **Plans:** Finish the installation of the ETI/Dalsa camera system with the 800 ft/min capability and begin the planned experimental program.

### III. PRINT WEB INSPECTION

- A. **Objective:** Develop a system for the off-line inspection of print cylinder verification materials.
- B. **Results:** Daedal has been chosen as the vendor to supply the material handling system. The motion control hardware will be integrated with an air chuck to hold the printed web in place during inspection.

An algorithm to create reference images for use in the cylinder verification inspection has been developed. This algorithm will provide a stable image for a wide range in the illuminating intensity. Work has begun on the inspection algorithm.

- C. **Plans:** Begin testing of different algorithms that may be used for the inspection process.

2022201542

**PROJECT NUMBER :** 6502  
**PROJECT TITLE :** Process Monitoring and Real Time Measurements  
**PROJECT LEADER :** K. B. Koller  
**PERIOD COVERED :** December, 1990

**I. GAS PHASE SMOKE ANALYSIS**

**A. Objective:** Complete installation of multicomponent gas-phase smoke analysis system for CTSD.

**B. Results:** A new system providing quantitative analysis of carbon monoxide, nitric oxide, hydrogen cyanide, and acetaldehyde in gas-phase MS smoke has been placed into routine operation in the Special Smoking Laboratory of CTSD. The analysis system consists of a new 5-port smoking machine interfaced to a Fourier Transform Infrared (FT-IR) spectrometer. The performance of the new system was evaluated and compared to that of the previously used analysis procedure. A second system has been installed, but not placed into routine operation. Personnel in CTSD have attended a training course in the operation and maintenance of FT-IR spectrometers.

**C. Plans:** Provide support to CTSD as needed.

**D. Reference:**

Joyner, B., "Gas Phase Analysis, FTIR Calibration", Memo to J. Garman, November 15, 1990.

**A. Objective:** Determine the effect of different puff volumes on MS and SS HCN deliveries.

**B. Results:** Experiments were conducted using IM13 cigarettes. Ten different puff volumes were used ranging from 4 mL to 70 mL. Two second duration puffs were taken once per minute. Both MS and SS HCN deliveries were affected by puff volume. MS HCN levels increased with puff volume whereas SS HCN levels were highest at the middle of the range of puff volumes used.

**C. Conclusions:** The dependence of HCN levels in gas-phase smoke on puff volume is attributed to variation in cigarette coal temperature.

**D. Reference:**

Parrish, M., PM Notebook 9034, pp. 21-22.

## II. R&D AND OPERATIONS SUPPORT

A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

The amount of ammonia was determined in samples submitted by J. Lephardt.

Deliveries of formaldehyde, acetaldehyde, acrolein, and acetone were determined in MS smoke for six cigarette samples submitted by R. Comes.

C. Plans: Continue to provide support as needed.

2022201544



**PROJECT NUMBER :** 6503  
**PROJECT TITLE :** Chemical Analysis  
**PROJECT LEADER :** B. M. Handy  
**PERIOD COVERED :** December, 1990

#### **I. ACETYLATED PULP**

- A. Objective:** Determine the percent acetyl content on handmade sheets of paper made from different batches of acetylated cellulose.
- B. Results:** A procedure provided by the USDA Forest Products Laboratory was applied to handmade paper sheets made from several batches of acetylated cellulose. Compared to the acetylated cellulose, the papers were lower in acetyl content than the corresponding starting material.
- C. Plans:** To measure the percent acetyl content on the "fines" from the paper making process.

#### **II. METHOPRENE KABAT**

- A. Objective:** Determine methoprene in tobacco from stemmeries to support Engineering efforts to assure that the application is within specifications.
- B. Results:** Methoprene on Dryer Exit and Hogshead Core samples from ULT Stemmary Qualification tests continue. Danville and Smithfield have been completed and Lexington is in progress. Build-up from apron and brushes at Richmond LPF were analyzed for methoprene. Kabat from Zoecon was assayed.
- C. Plans:** Continue support as needed.

#### **III. COOPERATIVE LEAF STUDIES**

- A. Objective:** To provide analytical support for the cooperative tobacco industry standards program.
- B. Results:** Alkaloids and sugars were determined on 399 tobacco samples from the 1990 S.C. Mammoth Tobacco Test and alkaloids, sugars and nitrogen on 160 flue-cured samples from the University of Florida Harvest Study.
- C. Plans:** Continue analytical support as required.

PROJECT NUMBER : 6505  
PROJECT TITLE : Special Investigations  
PROJECT LEADER : D. F. Ingraham  
WRITTEN BY : R. W. Kanipe  
PERIOD COVERED : December, 1990

**R&D AND OPERATIONS SUPPORT**

A. **Objective:** To provide analytical support to R&D and Operations personnel and projects.

B. **Results:**

An HPLC method was developed for the determination of propyl paraben in cigarette paper. The method was subsequently used to quantify propyl paraben in three paper samples for S. Baldwin.

Fourteen samples of Marlboro and Winston from the marketplace were analyzed for methoprene by HPLC. The results showed that Winston contained ~0.8 ppm methoprene and Marlboro contained ~0.4 ppm methoprene (in the samples analyzed).

An HPLC method was developed for the determination of nicotine in stack scrubber samples; twenty-eight samples were subsequently analyzed using this method. The limit of detection for this procedure was approximately 1 ppm.

Three capillary electrophoresis (CE) instruments were evaluated for purchase recommendations. This technique may be applicable for quantitation of flavors, tobacco constituents, smoke components, etc. that are not readily determined by GC or HPLC. While the theory of CE has been understood for the last decade, instrument development is still in its infancy. As a result, recommendations for purchase are pending new instrument introductions at the 1991 Pittsburgh Conference.

Acetic and butyric acids were determined in samples of Spanish Asta and Siva sheet material by capillary gas chromatography. These acids are being used to monitor spoilage.

**PROJECT NUMBER :** 6902  
**PROJECT TITLE :** Biochemical Special Investigations  
**PROJECT LEADER :** B. D. Davies  
**PERIOD COVERED :** December, 1990

**I. NICOTINE SPECIFIC MONOCLONAL ANTIBODY**

- A. Objective:** Develop a nicotine monoclonal antibody based enzyme-linked immunosorbent assay (ELISA) for the determination of nicotine concentrations in samples of interest to PM.
- B. Results:** Several experiments were conducted to examine the variation recently noted in absorbance values from our Bio-Tek microtiter plate reader. In one experiment, a nicotine inhibition curve was replicated in the four different quadrants of the plate. The plate was placed in the plate reader in the normal conformation and the absorbance of the individual wells determined. Then the plate was rotated 180° and the absorbances again determined. The data from this experiment are currently being reviewed and the results will be reported next month. In addition, a 1% triethanolamine (1% TEA) extraction of a Grizzel #2 Bright monitor blend was prepared and the nicotine content quantitated twice using a nicotine ELISA. The results of those assays suggested a problem with the plate reader. This was confirmed following an examination of the instrument by a technical consultant from Bio-Tek.
- C. Plans:** The Bio-Tek microtiter plate reader is currently being serviced. Upon its repair, conduct several analyses to assure the problems have been corrected. Conduct the final analysis of the overall variation of the nicotine ELISA.
- D. References:**
1. Crockett, E.A. Notebook 8998, pp. 160-165.
  2. Davies, B. D. Notebook No. 8638, pp. 193-196.

PROJECT NUMBER : 6906  
PROJECT TITLE : Biological Effects of Smoke  
PROJECT LEADER : G. J. Patskan  
PERIOD COVERED : December, 1990

I. JB6 MOUSE EPIDERMAL CELL TRANSFORMATION ASSAY (G. Nixon)

- A. Objective: To obtain a colony formation response to CSC treatment in soft agar, and to select a new serum stock lot.
- B. Results: Two soft agar assays testing the effects of 2R1 CSC and 12-O-tetradecanoylphorbol-13-acetate (TPA) in combination were completed. In both experiments, the combination of treatments resulted in lower colony counts than when TPA was used by itself. This may be a toxicity effect, as colony counts were lower in both promotion-sensitive and transformed cells when TPA and CSC were combined. Acetone was compared to dimethyl sulfoxide (DMSO) as a possible solvent for CSC in the soft agar assay. The two solvents were comparable in control plates (TPA), but neither was effective in supporting colony formation of the promotion-sensitive JB6 cells by CSC in soft agar. Two new lots of Hyclone serum were put into soft agar tests. The new lot which supported cell growth and colony formation in soft agar (using TPA treatment) most comparable to the current stock lot has been obtained in large quantities for use as the stock lot for the next year.
- C. Plans: The logistics of testing whole smoke in the soft agar assay will be studied.
- D. References:
- Burruss, T.J. Notebook No. 8896, p. 34.
- Vaughan, B.G. Notebook No. 8458, p. 109
- Nixon, G. M. Notebook No. 8711, p. 165.

II. SALMONELLA/MICROSOME (S/M) ASSAY

- A. Objective: To test the biological activity of experimental CSCs and other pertinent materials.
- B. Results: Several CSC samples were tested in support of the Crossed Solubles Base Web (CSBW) study. CSCs from experimental cigarettes prepared by individuals external to the Biochemical Research Division were also tested.
- Additionally, data was analyzed to evaluate a "screening" protocol for the S/M Assay.
- C. Plans: Test samples for biological activity as they become available. Document a "screening" protocol for the S/M Assay.

**D. References:**

Jones, R. Notebook No. 8769, p. 100.

Stagg, D. Notebook No. 9038, p. 49.

**III. PLANT TISSUE CULTURE (M. Shulleeta)**

- A. Objective:** To develop procedures for the establishment, maintenance and transformation of plant cell cultures.

- B. Results:** Studies were conducted to optimize both the salt concentration and osmoticum for purification, evacuation and culture of Burley 21 protoplasts. In preliminary experiments a complex medium (8P) with 0.33M sucrose as osmoticum was identified as an appropriate medium for use in tobacco cell transformation.

Microcalli were successfully generated from Burley 21 protoplasts even though these protoplasts had undergone spontaneous evacuation due to the suboptimal medium conditions used in a previous purification procedure. These microcalli have been plated onto standard callus proliferation medium following successive dilution into osmoticum-free medium.

Callus material generated from the routine cell culture of Burley 21 stem tissue has been transferred to regenerative medium to verify the capability of this cell line to form intact plantlets.

Burley 21 callus material cultured on a nicotine production medium (RM2) was tested via a Dragendorff squash-blot procedure to verify the presence of alkaloids in the tissue. The results indicated that alkaloids were produced by the RM2-grown callus in contrast to callus grown on standard proliferative medium which do not produce detectable levels of alkaloids.

- C. Plans:** Repeat Burley 21 protoplast purification and culture using 8P medium with 0.33M sucrose as osmoticum.

Assess the regenerability of Burley 21 calli cultured from evacuated and non-evacuated protoplasts.

Initiate suspension cultures of Burley 21 cells and determine the growth kinetics.

Electroporate Burley 21 protoplasts with PZO plasmid (coding for hygromycin resistance and GUS production) and assay for transient expression of GUS.

2022201549

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D. References:

Shulleeta, M. Notebook No. 8961.

2022201550

PROJECT NUMBER : 6908  
PROJECT TITLE : Smoke Condensate Studies  
PROJECT LEADER : A. H. Warfield  
WRITTEN BY : S. A. Haut  
PERIOD COVERED : December, 1990

## **I. REDUCTION OF MS TSNA BY THE REMOVAL OF TSNA PRECURSORS**

- A. **Objective:** To develop methods for the removal of TSNA precursors from filler.
- B. **Results:** TSNA analyses have been conducted on DBC burley, SCF-carbon dioxide extracted DBC burley, and the base webs derived from these fillers. For the burley tobacco, preformed TSNA levels are reduced 95% (NNN), 80% (NAT), 0% (NNK) and MS delivery reduced 41% (NNN), 52% (NAT), 35% (NNK). Filler nicotine is reduced >97% while minor alkaloids are virtually unchanged. TPM is reduced 17%. These results are not unusual.

Analysis of the base webs prepared from the above burleys was also completed. Preformed TSNA for the unextracted-burley base web was unusually high (two to ten-fold) when compared to 1986 burley base web or to 1986 typical base web from the R&D pilot plant. At present, there is no known cause for this anomaly. The preformed TSNA levels were very low in the SCF-burley base web as expected. No comparison between the two base webs is advisable until the apparent discrepancy in this 1990 burley base web is clarified. MS NNK for the two base webs is proportionally less dependent on preformed levels and exhibited a reduction of 39%. The analytical data for these base webs gives no indication how the TPM could be reduced to such an extent.

- C. **Plans:** Replicate base web TPMs and obtain gas-phase analyses on all four fillers.
- D. **References:**

Haut, S. A. Notebook No. 8891, p. 148.

## **II. REDUCTION OF MS TSNA BY INHIBITING THE PYROSYNTHESIZED TSNA**

- A. **Objective:** Reduce the levels of pyrosynthesized MS TSNA by blocking reaction pathways which form TSNA.
- B. **Results:** RLs from base web obtained from the R&D pilot plant this past year (1990 typical base web), CELs, and other components have been prepared and MS TSNA deliveries determined. In contrast to previous work which showed that addition of ascorbic acid, oriental-like sugar mixtures, and oriental/burley CEL mixtures reduced MS TSNA delivery for RL prepared with burley base web, no significant MS TSNA reductions were observed.

The filler TSNA behavior of the 1990 typical base web is unusual. Unlike burley and bright base webs observed in the past, there seemed to be a rapid and pronounced increase in preformed TSNA with age.

C. Plans: Some of the data will be replicated. A memo is being prepared.

D. References:

Tickle, M. H. Notebook No. 8936.

### III. ANALYTICAL PROCEDURES

A. Objective: To develop and maintain analytical methodology for TSNA or other compound classes where information is needed for determining relationships of TSNA to their precursors.

B. Results: Work continues to devise better collection and analysis methods for VNA and TSNA in aged and unaged smoke. Additional evaluations of the collection traps with the smaller pored frits continues with trials under more realistic and less ideal conditions. A minor change in the integrator program improved the differentiation between small peaks and baseline noise.

C. Plans: Continue refining and implementing method to actual collection environments.

D. References:

Morgan, W. R. Notebook No. 8905, p. 19.

### IV. MISCELLANEOUS AND SUPPORT STUDIES

A. Objective: To conduct studies of the TSNA content of filler and/or smoke and carry out other activities as necessary to support other PM programs.

B. Results: PAH determinations were conducted on a special sample along with 1R4F as control. Data has been collected in triplicate but final processing and interpretation is not yet complete.

Assistance was provided to Project 6906 in the collection of MS smoke. Modification of the standard procedure was required.

C. Plans: Complete PAH analysis and report results. Analyze samples in unextracted nicotine study.

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**D. References:**

Keene, C. K. Notebook No. 8946, p. 196.

Lambert, E. A. Notebook No. 8852, p. 185.

2022201553

PROJECT NUMBER : 6912  
PROJECT TITLE : Tobacco/Smoke Relationships  
PROJECT LEADER : R. R. Izac  
WRITTEN BY : S. Drew  
PERIOD COVERED : December, 1990

## **I. CROSS SOLUBLES BASE WEB (CHEMISTRY)**

A. **Objective:** To modify tobacco solubles and evaluate the smoke chemistry of fillers from various treated solubles and the appropriate base web.

B. **Results:** A study using ion exchange resins to remove salts and metal cations from tobacco solubles was continued. Burley CEL and BuS1 have been treated with a strong cation exchange resin to determine the appropriate starting material for use in future ion exchange studies.

Bright (Br), burley (Bu), and Oriental (MT) "solubles" and insolubles were freeze dried to determine the amount of solids present as part of a smoke chemistry study.

IT CSCs from the amino acid add-back, the 10K hollow fiber membrane and the Bu CEL stock comparison studies have been submitted for S/M assay testing.

Preparation and set-up of the Medimat® 220 electrodialysis (ED) unit was continued. Cation and anion cell membranes were assembled for use with the ED unit.

C. **Plans:** Continue to generate model fillers from various CSBW studies. The Br, Bu, and MT solubles and their corresponding insolubles will be applied to bright base web. Impaction trapped cigarette smoke condensate (IT CSC) generated from each model filler will be tested in the S/M assay.

### **D. References:**

1. Drew, S. Notebook No. 8950, pp. 81-83.
2. Hellams, R. Notebook No. 8959.
3. Zoller, M. Notebook No. 9005, pp. 85-93.

## **II. SUPPORT FUNCTION: SAMPLE PREPARATION**

A. **Objective:** To prepare chemical fractions and/or condensates as needed for biological and chemical analysis.

B. **Results:** A study to determine the biological and chemical differences in collection and/or processing methods was continued. IT CSC was collected on Cambridge filter pads at room temperature and in Elmenhorst cold traps (-78°C). Samples were prepared in DMSO and submitted for S/M testing. IT CSC was collected at ice water temperature for PAH and metal analyses.

A  $^{14}\text{C}$ -(U)-nicotine solution was added to aerated basin liquor and the solution exposed to bubbled oxygen in a reaction vessel. After 24 hours, the liquor was determined by HPLC to be depleted, or below the detection limit, of nicotine. Preliminary radioactivity results were obtained. Approximately 50% of the radioactivity was determined to be  $^{14}\text{CO}_2$ . The remainder of the activity was associated with a water soluble metabolite (45%) and the solids (5%) in the reaction mixture. The identity of the activity in the solids has not been determined.

C. **Plans:** Submit appropriate products to ARD for chemical analyses and to Project 6906 personnel for S/M testing.

D. **References:**

1. Hellams, R. Notebook No. 8959.
2. Izac, R. Notebook No. 8874, pp. 80-83.

2022201555

**PROJECT NUMBER :** 0008  
**PROJECT TITLE :** Computer Applications Division  
**WRITTEN BY :** John Blankinship, and John Palesis  
**PERIOD COVERED :** December, 1990

## **I. NEUROCOMPUTING**

**A. Objective:** Develop programming techniques for use of the HNC ANZA Plus Neurocomputer and Multilayer Backpropagation Neurosoftware. Investigate potential applications of neurocomputing to R&D problems, and apply where appropriate.

**B. Results:** Research continues into improved backpropagation training of multilayer networks, as applied to the cigarette analyticals vs. liking score mapping problem. Several difficulties have been encountered recently. The main problem is that different mappings (predictions) are obtained depending on the random seed used for weight initialization, although the mean squared errors are approximately the same. Other problems include getting stuck in quasi-local minima and extremely long training times.

Research into these problems is proceeding along several fronts, including: (1) allowing a few chances for recovery after the mean squared error has increased, rather than immediately decreasing the step size, (2) batching over the entire training set in certain situations, and (3) improved algorithms for controlling the learning rate and momentum factor.

**C. Plans:** Continue research.

## **II. Expert Systems Development**

**A. Objective:** Develop an Expert System for Cigarette Design.

**B. Results:** CigDES is built on top of two closely coupled subsystems: a mathematical cigarette model implemented and maintained in Fortran by Bill Dwyer and a model-based reasoning expert system implemented and maintained in Lisp and KEE by John Palesis. In view of the fact that the mathematical model is still evolving, it is necessary to establish a method whereby changes of this subsystem can be monitored and recorded so that they can be incorporated into the qualitative model and the model-based reasoning component of CigDES. We are presently studying how this can be accomplished with the UNIX System V Source Control System (SCCS). SCCS is a collection of UNIX system commands for controlling changes to files of text (storing and updating files, retrieving different versions, of files, recording who made each change and when it was made, etc.

- C. **Plans:** Updates of both the mathematical model Fortran files and the expert system Lisp files will be managed under SCCS.
- D. **References:** Palesis, J.A., Dwyer, R.W., Leister, D.L., and Kao, J.W., "Transforming Mathematical Product Evaluation Models Into Expert Systems for Product Design," Proceedings of the 3rd International Conference on Industrial & Engineering Applications of Artificial Intelligence and Expert Systems, pp. 404-415, 1990.

### III. Machine Learning

- A. **Objective:** Apply AI-based machine learning in scientific research.
- B. **Results:** We are presently using the machine induction tool 1st-Class to analyze data collected in a hydromagnesite study conducted by Ken Podraza. The primary objective is to discover any existing correlations between the extinction coefficient (visible sidestream reduction), on the one hand, and, on the other, paper weight, porosity, sizing (type of organic salts), %cation (how much sodium, magnesium, potassium, etc. is in the paper), and Standard Burn Time.

We have also written a technical report titled Machine Learning and Scientific Research: A Case for the ID3 Learning Algorithm./fR L2 "Plans" Present a technical seminar to demonstrate the use of the /fBID3/fR example-based learning algorithm in scientific research.

- C. **References:** (1) J. Palesis. AI-Based Machine Learning and Scientific Research. Memo to Colon Rowe, September 7, 1990. (2) J. Palesis. Machine Learning and Scientific Research: A Case for the ID3 Learning Algorithm. Technical Report, Philip Morris USA, December 21, 1990.

2022201557

PROJECT NUMBER : 8101  
PROJECT TITLE : Cigarette Testing Services  
WRITTEN BY : Joseph M. Garman  
PERIOD COVERED : January 1991

## **I. MARKET ACTIVITY**

**A. Objective:** To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

**B. Results:**

Domestic

R. J. Reynolds is distributing Camel Ultra Lights cigarettes in three packings: softpack - 84 mm (6.0 mg tar, 0.5 mg nicotine); box - 83 mm (5.9 mg tar, 0.5 mg nicotine); and 100 mm box (6.1 mg tar, 0.6 mg nicotine). These samples were received from Richmond and Arizona.

American is distributing American Lights 100 Menthol in Texas which delivers 11.0 mg tar and 0.9 mg nicotine.

Brown and Williamson is distributing Raleigh Extra King Size (16.8 mg tar, 1.0 mg nicotine); Raleigh Extra Lights King Size (11.6 mg tar, 0.8 mg nicotine) and Raleigh Extra Lights Menthol King Size (10.5 mg tar, 0.7 mg nicotine) in North Carolina.

Brown and Williamson is distributing Viceroy King Size Box (17.0 mg tar, 1.1 mg nicotine) and Viceroy Lights King Size Box (11.5 mg tar and 0.8 mg nicotine). Both of these cigarettes are 83 mm in length.

The cigarette paper on the following R. J. Reynolds brands are now 95-100% woodpulp.

- Century 85, 100
- Century Lights 85, 100
- Camel Ultra Lights 85, 100 soft pack & box
- Doral Full Flavor 85, 100 (plain & menthol)
- Doral Lights 85, 100
- Doral Ultra Lights 85, 100
- Magna 85 soft pack & box
- Magna Lights 85 soft pack & box
- Winston 80, 85, 100
- Winston Lights 80, 85, 100
- Winston Ultra Lights 85, 100

Cigarette papers on four recently introduced brands from Brown & Williamson consist of 95-100% woodpulp. This is the first indication of utilization of woodpulp in cigarette papers by Brown & Williamson. We will continue to monitor their brands.

- Raleigh Extra
- Raleigh Extra Lights
- Viceroy (Box)
- Viceroy Lights (Box)

#### International

No new brands were introduced during this period.

- C. Plans: Continue to monitor cigarettes introduced in the marketplace.

## II. SUGARS/ALKALOIDS LABORATORY

- A. Objective: To monitor and evaluate analytical technology.

- B. Results: The evaluation of the Alpkem autoanalyzer as a future replacement for the existing Technicon autoanalyzer system has been completed. The Alpkem system is a microsystem which uses micro-sized transmission and pump tubing, shorter dialyzers and less reagents. As a result, samples are analyzed in half the time of the Technicon system. Statistical evaluation of the initial data indicated that there is a statistical difference, but the magnitude of the difference is slight. Alkaloid data averaged a median sample difference of 0.1 mg higher on the Alpkem system. The sugars averaged a median sample difference of 0.3 mg lower on the Alpkem system. For both constituents the Alpkem results were within the established control limits on the Technicon system.

- C. Plans: Issue report on the results of this work.

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